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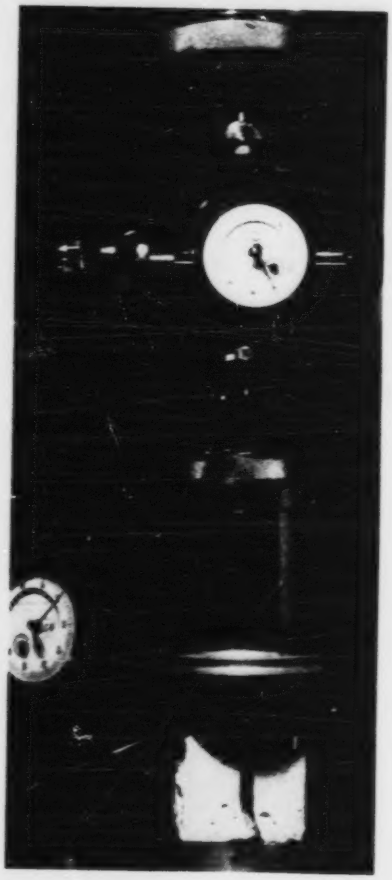

Investigation of Test Methods for Solidified Waste Evaluation - A Cooperative Program

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January 1991

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Investigation of Test Methods for Solidified Waste Evaluation - A Cooperative Program

by
J.A. Stegemann and P.L. Côté

Wastewater Technology Centre
Environment Canada

This program was conducted with the cooperation and support of
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Abstract

A study was undertaken by Environment Canada, in conjunction with the United States Environmental Protection Agency (U.S. EPA), Alberta Environment, and 15 industrial participants involved in developing or marketing solidification technology, to develop and validate 16 laboratory test methods for evaluating the physical and chemical properties of solidified wastes.

Environment Canada and U.S. EPA provided the 15 industrial participants with five untreated hazardous wastes. The industrial participants applied their proprietary processes to the wastes and returned the solidified products to four laboratories in Canada and the United States for testing. Seven physical tests, five leaching tests, and four micromorphological characterization methods were applied to the solidified products.

Although the reproducibilities of the different methods ranged from excellent to only fair, all of the methods were sufficiently reproducible to be valuable tools for evaluating the properties of a wide variety of solidified wastes.

The properties measured in the various tests show that the majority of solidified products are strong, durable materials of intermediate permeability, which immobilize heavy metal contaminants effectively, while providing little containment of organic compounds.

Résumé

Une étude a été entreprise par Environnement Canada, en collaboration avec l'Environmental Protection Agency (EPA) des États-Unis, l'Alberta Environment et 15 participants industriels oeuvrant dans le développement ou la commercialisation de techniques de solidification, afin d'élaborer et de valider 16 méthodes d'essai en laboratoire pour l'évaluation des propriétés physiques et chimiques des déchets solidifiés.

Environnement Canada et l'EPA ont fourni aux 15 participants industriels cinq déchets dangereux non traités. Les participants industriels ont appliqué leurs propres procédés aux déchets et ont renvoyé les produits solidifiés à quatre laboratoires au Canada et aux États-Unis pour fins d'essai. On a fait subir aux produits solidifiés sept essais physiques, cinq essais de lixiviation et quatre méthodes de caractérisation micromorphologique.

Bien que la reproductibilité des différentes méthodes ait varié, d'excellente à moyenne toutes les méthodes se sont avérées assez reproductibles pour constituer des outils précieux pour l'évaluation des propriétés d'une vaste gamme de déchets solidifiés.

Les propriétés mesurées lors des différents essais montrent que la plupart des produits solidifiés sont des matériaux résistants et durables, de perméabilité moyenne, qui immobilisent efficacement les métaux lourds contaminants, tout en retenant peu les composés organiques.

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Dofasco Incorporated
Harmon Environmental Services
McGill University
Peterson Contractors Inc.
The PQ Corporation
Solidtek Systems Inc.
Stablex Canada Inc.
Tricil Limited
United States Gypsum Company
Velsicol Chemical Corporation
West-Paine Laboratories Inc.

Executive Summary

Background

Landfilling is the disposal method of choice for the many hazardous wastes that are not recyclable, degradable, or combustible for practical or economic reasons. Solidification processes are designed to improve the physical and chemical characteristics of hazardous wastes to prevent toxic substances from contaminating the environment surrounding a landfill.

The numerous vendors of solidification processes make various claims as to the different degrees to which their treatment technologies affect the waste properties of concern. Hitherto, there has been no consistent evaluation procedure for solidified wastes. As a result, there has been little reliable information available regarding actual solidified waste properties.

Evaluating the efficacy of solidification processes is a problem faced by industry and regulators alike. A standard evaluation protocol is required to allow industry to develop and use effective solidification processes, and regulators to ensure adequate protection of the environment. More information regarding solidified waste properties is also required by regulatory agencies, to assist with setting guidelines and standards to protect the public.

In response to this need, a study was initiated by Environment Canada, in conjunction with the United States Environmental Protection Agency, Alberta Environment, and 15 American, Canadian, and European industrial participants involved in developing or marketing solidification technology, to develop and evaluate 16 laboratory test methods for characterizing the physical and chemical properties of a variety of solidified wastes. The results of this study are described in this report.

Objectives

The cooperative program had the following objectives:

1) to develop a set of laboratory test methods for characterizing the physical and chemical properties of a wide variety of solidified wastes

2) to evaluate the set of test methods by using real wastes and actual commercial solidification processes and by generating information regarding the reproducibility of the test methods

3) to initiate development of an international data base of properties of solidified wastes achievable with present

technology to assist regulatory agencies and the waste treatment industry in setting guidelines

4) to provide a basis for continued development of test methods and mathematical models

5) to promote international use of the test methods for uniform comparative evaluation of solidified wastes

Approach

Environment Canada and the United States Environmental Protection Agency provided the 15 industrial participants with samples of their choice of five fully characterized untreated hazardous wastes: two synthetic wastes containing heavy metals, an aluminum coil plating sludge, a dredged sediment, and a soil contaminated with wood preservation waste. The industrial participants applied their proprietary processes to the wastes and returned them for testing.

Seven physical tests and five leaching tests were applied to the solidified products by Environment Canada's Wastewater Technology Centre, the Alberta Environmental Centre, and the United States Army Engineers' Waterways Experiment Station. In general, each of the 69 solidified products was tested in duplicate in two of the three laboratories. In addition, four micromorphological characterization methods were applied to the solidified products for the two synthetic wastes and the wood preservation waste soil by Louisiana State University.

The test methods were chosen for characterization of the chemical and physical factors that affect the leachability of a waste form: 1) the degree of chemical immobilization of the contaminants (i.e., contaminant speciation and solubility under various conditions, and acid neutralization capacity), 2) the degree of physical immobilization of the contaminants (i.e., contaminant mobility in the solidified matrix), and 3) the potential for contact of groundwater with the waste (i.e., matrix hydraulic conductivity, strength, and weathering resistance). A number of physical parameters (bulk density, moisture content, and specific gravity), which provide basic information relevant to interpreting other tests, were also characterized. To maximize universal applicability of the tests, no attempt was made to simulate specific disposal conditions; the properties measured relate to the waste form and are independent of the disposal scenario.

Conclusions

With the exception of hydraulic conductivity and a specific gravity measurement technique, reproducibility of the physical tests was very good. For most of the physical tests, calculations using the intralaboratory variance showed that a minimum of four replicates is necessary to achieve an acceptable precision.

The results from the leaching tests showed that, although reproducibility was poor in the low concentration range, the tests are valuable tools for determining the effectiveness of a solidification process. A minimum of four replicates is also recommended for the leaching tests; however, depending on the desired application of the results, more replicates may be required to attain greater precision.

The properties measured in the physical and leaching tests show that the majority of solidified products are strong, durable materials of intermediate permeability, which immobilize heavy metal contaminants effectively, while providing little containment of organic compounds. The ranges of properties measured will be very useful to industry, in comparing the efficacy of various solidification options for their wastes, and to regulators in setting realistic guidelines for solidified waste properties.

The micromorphological techniques showed themselves to be more applicable as research tools for detailed examination of a few samples for well-defined features than as general methods for overall investigation of a large number of solidified products. The results showed that most of the solidified products were cement and/or pozzolan based.

Evaluation of the protocol in four leading laboratories has established a network of technical exchange, which will be invaluable in continued promulgation of the methods developed in this study, and in further cooperative work on the topic of solidified waste assessment.

Recommendations

Each of the test methods performed and analyzed in this study measures a different solidified waste property of interest. These solidified waste properties are significant only in the context of the proposed disposal environment, which must be considered when evaluating the results of the tests. As a follow-up to this study, a document that recommends performance criteria for four generic utilization and disposal scenarios has been prepared.

The test methods investigated in this study represent the state of the art at the time of the study's initiation. However, test method development is an evolutionary process; as technology and understanding of waste characteristics develop, development of these and other test methods should be continued.

In particular, continued development of test methods to examine contaminant availability, waste stability in environments of different oxidation/reduction potentials, immobilization of organic contaminants, and biodegradability is desirable. Further development of some of the tests investigated in this study is also required to improve their reproducibility and to allow comparison of these characteristics for different solidified products.

With regard to solidification processes, it appears from this study that the majority of processes investigated did not deal effectively with even relatively small quantities of organic contaminants in primarily inorganic waste types. Development of processes for enhanced containment of organic compounds is essential if this technology is to be used for site cleanup, or treatment of complex wastes with traces of organic contaminants, without pretreatment of the waste.

Section 1

Introduction

Landfilling is the disposal method of choice for the many hazardous wastes that are not recyclable, degradable, or combustible for practical or economic reasons. Solidification* processes are designed to improve wastes for land disposal by ameliorating their physical properties, immobilizing the contaminants to prevent groundwater contamination, or both. Usually, wastes considered for treatment by solidification contain heavy metals, such as cadmium and lead; historically, organic chemicals have been more difficult to immobilize effectively, but the number of processes addressing this problem is growing, particularly since many primarily inorganic wastes contain trace quantities of organic chemicals.

A typical solidified waste matrix is formed from cementing reactions, such as in portland cement, or pozzolanic reactions, such as those between lime and fly ash. Other materials used may include clays, polymers, or proprietary specialty sorbents (Pojasek, 1979; United States Army Engineers' Waterways Experiment Station, 1984; ASTM, 1989). The crucial properties of the matrix are its ability to retain contaminants over long periods of time and continued low permeability to groundwater.

The numerous vendors of solidification processes make various claims as to the different degrees to which their processes affect these properties of concern. Evaluating the efficacy of solidification processes is a problem faced by industry and regulators alike.

A common approach to evaluating the hazards posed by wastes is the attempted simulation of disposal site conditions (related to climate, geology, hydrology, etc.) with laboratory testing. This approach has a number of disadvantages:

1) It is difficult to simulate field conditions with laboratory-scale tests.

2) One test (or a small number of tests) cannot be applicable to many disposal site conditions.

3) It is impossible to simulate the effects of long periods of time using a short-term test.

4) No information concerning stabilization mechanisms is gained.

Another approach involves characterizing the properties of the solidified waste in terms of the chemical and physical factors affecting the leachability of the waste form (Côté et al., 1987a):

1) The degree of chemical immobilization of the contaminants, i.e., the contaminants may be chemically bound in a variety of compounds of varying solubilities and reactivities, or they may be physically adsorbed or trapped in the solidified waste matrix. The mechanism of immobilization affects the response of the waste to different groundwater characteristics, particularly with regard to pH.

---

* In the context of this report, the term "solidification" has been used in a collective sense to include processes sometimes termed fixation or stabilization.

2) The potential for contact of groundwater with the waste, i.e., the hydraulic conductivity of a waste, as well as that of its surroundings, determines whether soluble contaminants are transported through the solidified waste matrix by advection or diffusion. Resistance to weathering and compressive strength may also affect the surface area of waste in contact with the leaching groundwater.

This approach of solidified waste property determination has advantages corresponding to the disadvantages of the first approach:

1) Solidified wastes with particular characteristics can be matched to the disposal scenarios for which they are most suited.

2) The characteristics may be used as source terms in mathematical models for estimating the effects of long periods of time.

3) A better understanding of stabilization mechanisms can be gained.

A cooperative experimental program was initiated by Environment Canada, in conjunction with the United States Environmental Protection Agency (U.S. EPA), Alberta Environment, and 15 industrial participants, to develop laboratory-scale test methods for characterizing the physical and chemical properties of a solidified waste to assist the regulating and regulated communities in evaluating solidified waste products.

Twelve methods for measuring physical and chemical properties were assembled from other fields and were adapted to characterize solidified wastes at Environment Canada's Wastewater Technology Centre (WTC) and the Alberta Environmental Centre (AEC). Four techniques for micromorphological characterization were developed at Louisiana State University (LSU). These tests were applied to solidified products provided by the 15 industrial participants in the study by the Wastewater Technology Centre, the Alberta Environmental Centre, the United States Army Engineers' Waterways Experiment Station (WES), and Louisiana State University. The Waterways Experiment Station worked under an interagency agreement with the United States Environmental Protection Agency.

Although the selected test methods covered a wide range of solidified waste properties, they were not exhaustive. The methods represent the state of the art at the time of the study's inception in 1985. Development of these and other tests has continued since then, and is encouraged.

1.1 Objectives

The cooperative program had the following objectives:

1) to develop a set of laboratory test methods for characterizing the physical and leaching properties of a wide variety of solidified wastes

2) to evaluate the set of test methods by using real wastes and actual commercial solidification processes and by generating information regarding the reproducibility of the test methods

3) to initiate development of an international data base of properties of solidified wastes achievable with present technology to assist regulatory agencies and the waste treatment industry in setting guidelines

4) to provide a basis for continued development of test methods and mathematical models

5) to promote international use of the test methods for uniform comparative evaluation of solidified wastes

It was not the purpose of this study to identify a regulatory testing protocol or to set performance criteria.

1.2 Approach

The numerous study participants and their responsibilities are listed in Table 1.

Five raw wastes were chosen for use in the study based on suggestions from all participants (see Section 2.1). The wastes were selected to include wastes containing problem contaminants, routinely solidified contaminants, or both.

Table 1 Participants and Their Responsibilities

Environment Canada Industrial Programs Branch	Project management
Industrial participants (see Appendix A)	Treatment of wastes by solidification
Environment Canada Wastewater Technology Centre	Assistance with project management Test method development Assistance with raw waste collection and characterization Testing of raw and solidified wastes Data reduction Final report preparation
Alberta Environmental Centre	Test method development Assistance with raw waste characterization Testing of solidified wastes Assistance with final report
United States Army Engineers' Waterways Experiment Station	Assistance with raw waste collection and characterization Testing of solidified wastes Peer review of final report
Louisiana State University	Microstructural and microchemical (LSU) characterization of selected solidified products
United States Environmental Protection Agency Office of Solid Waste	Support for Dynamac Corporation Support for Radian Corporation Peer review of final report
United States Environmental Protection Agency Risk Reduction Engineering Research Laboratory	Support for Waterways Experiment Station Peer review of final report
Radian Corporation	Assistance with raw waste collection and characterization
Dynamac Corporation	"Third party" action as intermediary between industrial participants and testing laboratories to protect proprietary information

A 20 L pail of each raw waste and information regarding its chemical and physical characteristics were sent to each of the 15 industrial participants at a rate of approximately one waste per month. After allowing 1 month for experimentation, solidification kits, containing specimen molds and preparation guidelines, were sent to those industrial participants interested in treating the waste.

After solidification by an industrial participant's desired process, the solidified material was returned to the laboratories for testing. All interaction between the industrial participants and the testing laboratories took place through the action of a third party. The companies participated under the agreement that their identities would remain unconnected with their specific solidified products unless they chose otherwise. It was the task of Dynamac Corporation to ensure the confidentiality of information proprietary to the industrial participants. Dynamac Corporation relabelled the product of each industrial participant with a unique secret code and distributed the randomized samples to the laboratories for testing.

Testing was performed at the Wastewater Technology Centre, the Alberta Environmental Centre, the United States Army Engineers' Waterways Experiment Station, and Louisiana State University. The Wastewater Technology Centre, Alberta Environmental Centre, and Waterways Experiment Station applied the protocol of 12 test methods to the solidified products such that, in general, duplicates of each test were carried out for each solidified product in two laboratories (see Section 4.1). Louisiana State University performed microstructural and microchemical characterizations of the solidified products for three of the raw waste types.

When testing was complete, analysis and interpretation of results were carried out at the Wastewater Technology Centre, with the assistance of the other participants. Each industrial participant was given the opportunity to examine the data for their solidified products and to choose to have their products identified with their corporate name (see Appendix A).

Wastes

2.1 Raw Wastes

Five raw wastes, including a synthetic solution, a synthetic sludge, an aluminum coil plating sludge, dredged sediment, and a wood preservation waste contaminated soil (Table 2), were collected from their generators, mixed, and each divided into fifteen 20 L pails. A composite sample of each waste was characterized by the collecting laboratory. Based on this analysis, as many as seven contaminants of interest were chosen for each waste, to be monitored in further testing. Composite samples were then analyzed for the contaminants of interest by two other laboratories. In addition, four aliquots were taken at random from the 15 pails and analyzed by the collecting laboratory to determine sample homogeneity; the 15 pails were then distributed to the industrial participants. Table 2 summarizes the analytical methods used.

For each contaminant in each waste, the concentrations determined in the above-mentioned analyses were plotted on normal probability paper to isolate outliers (Chambers et al., 1983). This technique also demonstrated that, in general, concentrations measured in the composites by different laboratories belonged to the same population as concentrations measured in the four different aliquots, i.e., the variability caused by waste heterogeneity was within analytical variability. In addition, interlaboratory variability was similar to intralaboratory variability, except in the case of the Waterways Experiment Station heavy metal hydroxide sludge. Average concentrations and coefficients of variation were calculated for each waste and contaminant, using all of the measured concentrations that appeared to belong to the same population, regardless of the laboratory or the type of sample (i.e., aliquot or composite). Outliers indicated by the probability plots were confirmed using the Q test (Skoog and West, 1974). Tables 3 and 4 summarize the characteristics of each raw waste that were used in further calculations throughout the report. In some cases, the analytical variability was very high. A brief discussion of each waste follows.

2.1.1 WTC Synthetic Solution

A solution containing 0.04 M of each of $CrCl_3 \cdot 6H_2O$, $Cd(NO_3)_2 \cdot 2H_2O$, $Pb(NO_3)_2$, $NaAsO_2$, and phenol in distilled water was used in previous studies at the Wastewater Technology Centre (Côté and Hamilton, 1983) and was included in the cooperative program. The solution had a total dissolved solids content of 3.4% and a bulk density of 1.0 g/cm^3. Its pH was 2.5. The waste was prepared by Radian Corporation. Table 3 also lists the calculated contaminant concentrations added. The average measured contaminant concentrations were 2400 mg of arsenic, 4600 mg of cadmium, 1600 mg of chromium, 8100 mg of lead, and 3700 mg of phenol per kilogram of solution. The percent differences* between the calculated and measured concentrations of these contaminants were -20% for arsenic, 1% for cadmium, -25% for chromium, -3% for lead, and -2% for phenol. The measured concentrations were used in further calculations. No outliers were isolated from the populations of nine analyses for arsenic,

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* Percent difference = 100 x (measured value - calculated value)/calculated value.

Table 2 Extraction Methods for Raw Waste Characterization

Waste	Contaminants of Interest	Extraction Method
WTC synthetic solution	Arsenic	Sulphuric/nitric digestion[a]
	Cadmium	Aqua regia/H_2O_2 digestion[a]
	Chromium	Aqua regia/H_2O_2 digestion
	Lead	Aqua regia/H_2O_2 digestion
	Phenol	4AAP method[b]
WES synthetic sludge	Cadmium	Aqua regia/H_2O_2 digestion
	Chromium	Aqua regia/H_2O_2 digestion
	Nickel	Aqua regia/H_2O_2 digestion
	Mercury	V_2O_5 hot block/cold vapour method[a]
Aluminum coil plating sludge	Arsenic	Alkaline fusion[a]
	Aluminum	Aqua regia/H_2O_2 digestion
	Barium	Aqua regia/H_2O_2 digestion
	Cadmium	Aqua regia/H_2O_2 digestion
	Calcium	Aqua regia/H_2O_2 digestion
	Chromium	Aqua regia/H_2O_2 digestion
	Copper	Aqua regia/H_2O_2 digestion
	Iron	Aqua regia/H_2O_2 digestion
	Lead	Aqua regia/H_2O_2 digestion
	Nickel	Aqua regia/H_2O_2 digestion
	Thallium	Aqua regia/H_2O_2 digestion
	Cyanide	Alkaline distillation[a]
Dredged sediment	Arsenic	Alkaline fusion
	Aluminum	HF digestion[a]
	Cadmium	HF digestion
	Chromium	HF digestion
	Copper	HF digestion
	Iron	HF digestion
	Lead	HF digestion
	Manganese	HF digestion
	Nickel	HF digestion
	Zinc	HF digestion
	Mercury	V_2O_5 hot block/cold vapour method
	Cyanide	Alkaline distillation
	Polychlorinated biphenyls (PCBs)	WTC GC method[a] or AEC GC method (PCB)[c] or U.S. EPA method 8080 (GC)[d]
	Polyaromatic hydrocarbons (PAHs)	U.S. EPA method 8270 (GCMS)[d]
Wood preservation waste soil	Arsenic	Alkaline fusion
	Aluminum	HF digestion
	Antimony	HF digestion
	Barium	HF digestion
	Cobalt	HF digestion
	Calcium	HF digestion
	Chromium	HF digestion
	Copper	HF digestion
	Iron	HF digestion
	Lead	HF digestion
	Nickel	HF digestion
	Selenium	HF digestion
	Silver	HF digestion
	Silicon	HF digestion
	Thallium	HF digestion
	Vanadium	HF digestion
	Mercury	V_2O_5 hot block/cold vapour method
	Pentachlorophenol (PCP)	U.S. EPA method 8040 (GC)[d] or AEC GC method (PCB)[c]
	Polyaromatic hydrocarbons	AEC HPLC method (PAH)[c] or U.S. EPA method 8270 (GCMS)

[a] Source: WTC (1985).

[b] Source: APHA (1981).

[c] Source: AEC (1985b).

[d] Source: U.S. EPA (1980).

Table 3 Summary of Contaminant Concentrations in the Raw Wastes

Waste	Contaminant	Average (mg/kg, w/ww) Calculated	Average (mg/kg, w/ww) Measured	Range	CoV (%)	n	N
WTC synthetic solution	Arsenic	3 000	2 400	1 345 to 4 130	36	9	9
	Cadmium	4 500	4 559	4 069 to 5 172	7	9	9
	Chromium	2 100	1 567	1 360 to 1 800	10	9	9
	Lead	8 300	8 051	7 400 to 8 585	6	8	9
	Phenol	3 800	3 719	3 341 to 4 029	6	7	8
WES synthetic sludge	Cadmium		3 984	3 325 to 4 810	-	-	8
	Chromium		18 439	15 000 to 21 749	-	-	8
	Nickel		19 187	17 000 to 22 130	-	-	8
	Mercury		233	35 to 499	-	-	8
Aluminum coil plating sludge	Arsenic		11	0.2 to 26.5	92	10	11
	Chromium		1 030	960 to 1 062	5	4	8
	Lead		43	2.3 to 104	75	11	11
	Thallium		17	10.3 to 28	47	6	7
	Total cyanide		1 793	1 125 to 2 766	31	12	12
Dredged sediment	Chromium		219	88 to 343	45	9	9
	Copper		72	50 to 79	12	9	9
	Lead		145	97 to 209	28	9	9
	Mercury		0.7	0.03 to 1.3	92	9	9
	Zinc		943	579 to 1 373	28	9	9
	Polychlorinated biphenyls		6.6	1 to 13	71	9	9
	Total organic carbon		90 000	85 000 to 98 000	8	3	4
Wood preservation waste soil	Arsenic		71	1.8 to 176	80	10	10
	Lead		68	24 to 98	56	10	10
	Mercury		0.02	0.02 to 0.03	25	3	4
	Pentachlorophenol		11 213	2 387 to 18 576	47	8	8
	Polyaromatic hydrocarbons		308	246 to 409	19	7	8

Note: n, number of measurements used in calculating the average.
N, total number of measurements.
N - n, number of outliers.
CoV, coefficient of variation; CoV = 100 x standard deviation/average.
For the Waterways Experiment Station synthetic sludge, the measured average was calculated using the average measurement in each of the three laboratories; CoV and n have not been shown, as they are not comparable.

Table 4 Additional Raw Waste Characteristics

Waste	Parameter	Average	CoV (%)	N
WTC synthetic solution	Bulk density (g/cm^3)	1.0	<1	4
	Total solids (%, w/ww)	3.4	8	5
	pH	2.5	-	-
WES synthetic sludge	Bulk density (g/cm^3)	1.2	-	2
	Total solids (%, w/ww)	26	-	13
	pH	11	-	-
Aluminum coil plating sludge	Bulk density (g/cm^3)	1.2	<1	3
	Total solids (%, w/ww)	28	10	4
	pH	7	-	-
Dredged sediment	Bulk density (g/cm^3)	1.3	6	8
	Total solids (%, w/ww)	39	7	7
	pH	7	-	-
Wood preservation waste soil	Bulk density (g/cm^3)	1.1	-	3
	Total solids (%, w/ww)	94	-	2
	pH	5	-	-

Note: N, total number of measurements, which in this case equals the number of measurements used in calculating the average.
CoV, coefficient of variation; CoV = 100 x standard deviation/average.

cadmium, and chromium; however, there was one analytical outlier for lead and one for phenol.

2.1.2 WES Synthetic Sludge

A synthetic heavy metal sludge was used in previous work at the Waterways Experiment Station to investigate interferences in solidification processes (Cullinane et al., 1987). This waste was also provided to the industrial participants in the cooperative study. The waste was prepared by treating a solution containing 600 times the United States Environmental Protection Agency extraction procedure limit of cadmium, chromium, nickel, and mercury nitrates with calcium hydroxide to precipitate the metals. The resulting 8% solids hydroxide sludge was dewatered to 26% solids. The pH was 11. Although the sludge was prepared synthetically, contaminant concentrations could not be calculated accurately because a complete mass balance for the complex sludge preparation process was not available. Average measured concentrations of the contaminants in the final sludge were 4000 mg of cadmium, 18 000 mg of chromium, 19 000 mg of nickel, and 200 mg of mercury per kilogram of sludge. Normal probability plots of the concentrations measured in the various samples of this waste showed that the multiple analyses conducted at the Waterways Experiment Station displayed less variability than was observed between the three different laboratories. To avoid calculating average contaminant concentrations that would be weighted in favour of the Waterways Experiment Station analyses, the total average was calculated from average concentrations in each laboratory.

2.1.3 Aluminum Coil Plating Sludge

Centrifuged clarifier underflow sludge from an aluminum coil plating facility was obtained by Radian Corporation. This sludge was produced by adding lime to precipitate metals from acidic rinse waters, and ferrous sulphate was utilized to precipitate cyanide as iron cyanide complexes. The sludge had a total solids content of 28% and a bulk density of 1.2 g/cm^3. The pH was 7. The average concentrations of the contaminants of interest were 10 mg of arsenic, 1000 mg of chromium, 40 mg of lead, 20 mg of thallium, and 1800 mg of total cyanide per kilogram of sludge. No outliers were isolated for cyanide; one outlier was apparent for each of arsenic and thallium. Normal probability plotting of all measured chromium concentrations showed two distinct populations. Based on concentrations measured in the solidified waste samples, it appeared that the population with the lower mean represented the raw waste most accurately. All numbers in the population with the higher mean (3900 mg/kg) were measured at a considerably later time period; air drying of the sample may have taken place in the meantime, resulting in erroneously high measurements. The high numbers were not used in further calculations.

2.1.4 Dredged Sediment

Dredged sediment from a heavily industrialized harbour was selected for inclusion in the cooperative program since solidification is often considered as a treatment option for contaminated sediments. It was obtained by the Wastewater Technology Centre and homogenized manually by coning and quartering. The dredged sediment had a bulk density of 1.3 g/cm^3. The total solids content was 39% and the pH was approximately 7. The following average contaminant concentrations were obtained from analysis of composites and aliquots: 200 mg of chromium, 70 mg of copper, 150 mg of lead, 0.7 mg of mercury, 900 mg of zinc, and 7 mg of polychlorinated biphenyls per kilogram of sediment. The dredged sediment also contained approximately 9% total organic carbon, which was monitored in some of the leach tests, although it was probably not a result of organic pollutants, but naturally occurring organic matter. Plotting the measured concentrations for each contaminant on normal probability paper did not isolate outliers for any of the contaminants, but one outlier was detected among the measurements of total organic carbon, which was confirmed by the Q test.

2.1.5 *Wood Preservation Waste Soil*

Soil contaminated with wood preserving solution is a difficult waste to treat because the concentration of organic compounds is not high enough to economically justify incineration, and organic chemicals are difficult to immobilize using solidification. The contaminated soil obtained by Radian Corporation for investigation in the cooperative program contained primarily pentachlorophenol, with smaller quantities of other wood preserving agents, including heavy metals and polyaromatic hydrocarbons. This waste had a solids content of 94% and a bulk density of 1.1 g/cm^3. It was mixed manually by coning and quartering. It contained the following average contaminant concentrations: 70 mg of arsenic, 70 mg of lead, 0.02 mg of mercury, 11 000 mg of pentachlorophenol, and 300 mg total polyaromatic hydrocarbons per kilogram of soil. Phenanthrene appeared to be the main polyaromatic hydrocarbon detected. One outlier was isolated in the normal probability plot of mercury concentrations and confirmed by the Q test. No outliers were apparent in the plots for the other contaminants.

2.2 *Solidified Wastes*

The shipment of each raw waste to the industrial participants was followed by solidification kits containing molds and instructions for specimen preparation. The instructions for specimen preparation included directions for molding and curing samples, and a record sheet, which the industrial participants completed with very general process details and returned to the testing laboratories with their samples. As previously mentioned, the raw wastes were solidified by the industrial participants' proprietary processes. Each solidified product was identified only by a unique secret code during testing. Appendix A contains any additional information that the industrial participants chose to release concerning their products.

The industrial participants were instructed to prepare only one batch of solidified waste for each raw waste to avoid introducing another source of variability between samples, but this was not feasible in some instances because of equipment limitations.

Specimens were cured within their molds, sealed in plastic bags to prevent moisture loss. Testing was originally planned to begin after 56 days of curing, to allow sufficient time for slow cementing pozzolanic reactions to occur. This target time period varied in practice, although most samples were cured for at least 56 days prior to unconfined compressive strength measurement. Most testing of the more time independent properties was carried out in the month following unconfined compressive strength measurement, although laboratory capacity limitations resulted in backlogging of some samples for up to a year. The dates for solidification and the beginning of testing are summarized in Table 5.

Weight change factors for each solidification process were calculated using the information provided on the solidification process record sheet. These are summarized in Table 6.

The weight change factors were used with the average contaminant concentrations in the raw wastes to calculate contaminant concentrations in the solidified products; the concentration differences between solidified products were small in comparison with analytical variability. In a few cases, liquid phases were produced during the solidification process, which were chemically analyzed at the Wastewater Technology Centre. The calculated concentrations in the corresponding solidified products were adjusted for the amount of contaminant that remained in the liquid phase. Some of the solidified products were analyzed for the contaminants of interest using the same analytical methods that were used for the raw wastes, for comparison with the calculated

Table 5 **Ranges of Solidification Dates**

Raw Waste	No. of Products	1985										1986								
		Aug. 16	30	Sept. 13	27	Oct. 1	25	Nov. 8	22	Dec. 6	20	Jan. 3	17	31	Feb. 14	28	Mar. 14	28	Apr. 1	25
WTC synthetic solution	14		<---	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	--->		+
WES synthetic sludge	14											<---	-----	-----	-----	-----	--->	+		
Aluminum coil plating sludge	9							<---	-----	--->		+								
	4								<---	-----	-----	-----	-----	-----	-----	-----	-----	-----	--->	+
Dredged sediment	8				<---	-----	--->			+										
	7				<---	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	--->	+
Wood preservation waste soil	13										<---	-----	-----	--->	+					

Note: - - - - -, range of solidification dates.
+, start of testing.

Table 6 **Weight Change Factors for Solidified Products**

Raw Waste	Weight Change Factor* (median)	Range
WTC synthetic solution	2.35	1.36 to 4.58
WES synthetic sludge	1.83	1.20 to 4.66
Aluminum coil plating sludge	1.68	1.20 to 3.72
Dredged sediment	1.53	1.14 to 3.18
Wood preservation waste soil	2.20	1.20 to 6.04
Overall	1.79	1.14 to 6.04

* Mass of solidified waste/mass of raw waste.

contaminant concentrations. The percent differences between calculated and average measured values are summarized in Table 7, and compared with the coefficients of variation for chemical analysis of the contaminant in the raw waste. A percent difference of magnitude greater than twice the coefficient of variation suggests that a real difference exists between the calculated and measured concentrations. Aside from analytical error, such a difference may be explained by one of the following:

1) a matrix effect could have caused reduced analytical recovery from the solidified waste compared with the raw waste or, conversely, matrix interference could result in enhancement of the measurement

2) solidification additives or pretreatment may not have been reported correctly

3) the contaminant may have been present in one of the solidification additives

4) volatilization of organic contaminants may have taken place

For virtually all contaminants, the difference between calculated and measured concentrations exceeded the variability of the raw waste for some solidified products. The difference between the calculated and measured numbers is likely a result of matrix effects in most cases.

A brief discussion of the solidified products for each raw waste follows.

Table 7 Percent Differences Between Measured and Calculated Contaminant Concentrations in the Solidified Wastes

Raw Waste	Contaminant	CoV (%) for Raw Waste Analyses	Range of Percent Differences	Number of Percent Differences <2CoV	Number of Products Analyzed
WTC synthetic solution	Arsenic	36	-	-	0
	Cadmium	7	-34 to 68	8	14
	Chromium	10	-10 to 433	5	14
	Lead	6	-48 to 19	2	14
	Phenol	6	-100 to 8	3	12
WES synthetic sludge	Cadmium	-	-16 to 109	-	-
	Chromium	-	-13 to 118	-	-
	Nickel	-	-10 to 115	-	-
	Mercury	-	-50 to 89	-	-
Aluminum coil plating sludge	Arsenic	92	-82 to 1049	6	8
	Chromium	5	-42 to 126	2	12
	Lead	75	-78 to 719	7	12
	Thallium	47	-90 to -52	8	8
	Total cyanide	31	-82 to 45	9	10
Dredged sediment	Chromium	45	-12 to 142	8	9
	Copper	12	39 to 137	0	9
	Lead	28	0 to 118	2	9
	Mercury	92	-66 to 349	3	4
	Zinc	28	-21 to 23	9	9
	Polychlorinated biphenyls	71	-	-	0
	Total organic carbon	8	-	-	0
Wood preservation waste soil	Arsenic	80	-	-	0
	Lead	56	-	-	0
	Mercury	25	-	-	0
	Pentachlorophenol	47	-98 to 22	10	13
	Polyaromatic hydrocarbons	19	-	-	0

Note: CoV, coefficient of variation; CoV = 100 x standard deviation/average.
Percent difference = 100 x (measured value - calculated value)/calculated value.

2.2.1 *WTC Synthetic Solution*

Fourteen of the 15 industrial participants chose to solidify the Wastewater Technology Centre synthetic solution. The median weight change factor across the different solidified products was 2.35. The weight change factors ranged from 1.36 to 4.58. Two industrial participants submitted liquid phases with their solidified products. These were analyzed and found to contain less than 1 mg of each of arsenic, cadmium, chromium, and lead per kilogram of liquid, but several hundred milligrams of phenol per kilogram of liquid. A third liquid phase was reported, but not submitted for analysis; the volume was very small.

No comparison between calculated and measured concentrations in the solidified products was made for arsenic; however, comparisons were made for cadmium, chromium, lead, and phenol. Chromium concentrations measured in the products were consistently higher than expected, whereas lead concentrations were consistently lower. Measured phenol concentrations were also consistently lower than calculated. Volatilization of phenol may have occurred, and in some cases the aqueous synthetic waste may have been pretreated to remove phenol, although no such pretreatment was reported.

Unforeseen circumstances resulted in a wide range of solidification dates. Consequently, curing times between the date of solidification and the determination of unconfined compressive strength (start of testing) ranged from 4 to 33 weeks. Eleven of the 14 solidified products experienced the recommended minimum curing period of 56 days.

2.2.2 *WES Synthetic Sludge*

The Waterways Experiment Station synthetic sludge was also solidified by 14 of the 15 industrial participants. Weight change factors ranged from 1.20 to 4.66, with a median of 1.83. A liquid phase was submitted with one solidified product and was found to contain less than 1 mg of each of the contaminants of interest per kilogram of liquid.

Percent differences between calculated and measured concentrations in the solids ranged from -16 to 62 for cadmium, -13 to 59 for chromium, -10 to 69 for nickel, and -50 to 89 for mercury. These ranges do not include the percent differences for two solidified products for which the percent differences were around 100 for cadmium, chromium, and nickel, suggesting that the sludge may have been dewatered before solidification.

Curing times before the start of testing ranged from 2 to 11 weeks for the synthetic sludge solidified products.

2.2.3 *Aluminum Coil Plating Sludge*

Thirteen of the 15 industrial participants applied their solidification processes to the aluminum coil plating sludge. Weight change factors ranged from 1.20 to 3.72, with a median of 1.68. No liquid phases were submitted for any of the solidified products.

Comparison of calculated and measured contaminant concentrations in the raw wastes showed that measured lead concentrations tended to be higher, whereas measured thallium concentrations were lower.

Nine solidified products were received by the laboratories in time for the initial testing deadline. The curing times before the start of testing of these products ranged from 6 to 9 weeks. The remaining four solidified products were received late for the initial testing deadline and were tested at a later date. The curing periods for these solidified products ranged from 2 to 21 weeks.

2.2.4 *Dredged Sediment*

All 15 of the industrial participants treated the dredged sediment. The median weight change factor was 1.53, with weight change factors ranging from 1.14 to 3.18. One industrial participant submitted a liquid phase for this waste. Contaminant concentrations in the liquid phase were less than 1 mg per kilogram of liquid, except for zinc, which was 2.5 mg per kilogram of liquid.

In comparisons of measured and calculated contaminant concentrations in the solidified products, measured copper and lead concentrations were consistently higher than calculated concentrations. Analysis of zinc alone did not appear to be influenced by the presence of the solidified waste matrix. No comparison was carried out for polychlorinated biphenyl concentrations.

Eight of the solidified products were received before the initial testing deadline. These products experienced curing times before unconfined compressive strength testing ranging from 6 to 9 weeks. The remaining seven solidified products, which were received and tested late, experienced curing periods ranging from 2 to 29 weeks.

2.2.5 Wood Preservation Waste Soil

The wood preservation waste soil was solidified by 13 industrial participants. The solidified products had weight change factors ranging from 1.20 to 6.04, with a median of 2.2. No liquid phases were produced for this waste.

No comparison between measured and calculated concentrations in the solidified products was carried out for arsenic, lead, mercury, or polyaromatic hydrocarbons. Pentachlorophenol concentrations in the solidified wastes were underestimated by analysis at the Alberta Environmental Centre compared with the calculated concentrations.

Curing periods varied from 2 to 9 weeks before the start of testing.

Section 3

Test Methods for Solidified Waste Evaluation

The test methods applied to the solidified products included seven physical tests, five chemical tests, and four micromorphological characterization techniques, as listed in Table 8.

The sources of the tests are diverse. Wherever possible, existing tests were adapted for the purpose of determining intrinsic solidified waste properties. A preliminary interlaboratory reproducibility study was carried out by the Alberta Environmental Centre, Wastewater Technology Centre, and Waterways Experiment Station to compare laboratory performances and resolve inconsistencies in the test methods (AEC, 1985a). Each test is summarized below.

3.1 Physical Evaluation Methods

Bulk density, specific gravity, and moisture content are engineering properties. They are used to determine the volume change factor for a solidification process, which is an important cost consideration. They are also used in porosity calculations, and may potentially be correlated with other properties of environmental concern, although such analysis was beyond the scope of this report.

Hydraulic conductivity, unconfined compressive strength, and freeze/thaw and wet/dry weathering resistance relate to the physical integrity of the solidified waste.

3.1.1 Bulk Density

Bulk density measurement is carried out by weighing a specimen of known volume and

Table 8 Test Methods for Solidified Waste Evaluation

Physical tests:	Bulk Density Specific Gravity Water Content Hydraulic Conductivity Unconfined Compressive Strength Freeze/Thaw Weathering Wet/Dry Weathering
Chemical tests:	Equilibrium Extraction U.S. EPA Toxicity Characteristic Leaching Procedure Acid Neutralization Capacity Sequential Chemical Extraction Dynamic Leach Test
Micromorphological techniques:	X-ray Diffraction Scanning Electron Microscopy (with Energy Dispersive X-ray Analysis) Optical Microscopy

reporting the mass per unit volume (g/cm^3). For accuracy, one test consists of three replicates.

3.1.2 *Specific Gravity*

Specific gravity is the mass of the dry solid portion of the waste per mass of an equivalent volume of water. Two techniques were used to determine this property.

The first is a wet method whereby the volume of a sample of known mass is determined by water displacement of the pore spaces in a volumetric flask. The test applied in this study is an adapted version of the American Society for Testing and Materials (ASTM) Standard Test Method for Specific Gravity of Soils (D 854-58). Immersion of the sample in water has the potential to dissolve the sample matrix, which would result in a false volume measurement. For this reason, a dry method was also carried out. Helium is used to fill the pore spaces of the sample of known mass, without dissolving the sample matrix. Both tests consist of three replicates.

3.1.3 *Moisture Content*

The method used to determine the moisture content was a simplification of the ASTM Standard Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil Aggregate Mixtures (D 2216-80). The mass of a sample of crushed solidified waste is determined before and after drying in a gravity convection oven. Drying is carried out at 60°C in an attempt to measure moisture content in excess of the water of hydration. The moisture content is reported as the mass of water per mass of wet sample. Again, one test consists of three replicates.

3.1.4 *Hydraulic Conductivity*

Hydraulic conductivity is a solidified waste's permeability to water, expressed in metres per second. The hydraulic conductivity of a solidified waste, in relation to that of the surrounding material, controls the flow of groundwater around or through the waste in a landfill; the groundwater flow regime, in turn, determines whether leaching occurs by diffusion or advection (Côté and Bridle, 1987).

Hydraulic conductivity was determined by a falling-head method. A 7.62 cm cylindrical specimen is confined by a pressurized membrane in a triaxial cell to prevent channeling (similar to the Draft ASTM Standard Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using Flexible-wall Parameters). The sample is saturated and saturation is verified by measuring the time required to transmit a compression wave through the sample. Tap water is allowed to flow vertically through the sample, and the hydraulic conductivity is calculated by Darcy's law from the time required to produce a certain volume of flow. The method follows saturation by immediate hydraulic conductivity measurement, without attempting to achieve steady-state conditions.

3.1.5 *Unconfined Compressive Strength*

Unconfined compressive strength is a measure of the ability of a solidified product to resist mechanical stresses such as might be imposed in a landfill by overburden or heavy land-moving equipment. Disintegration of a monolithic waste form under such stresses results in greater contact with groundwater.

The unconfined compressive strength test is adapted from the ASTM Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (C 109-80). A 5.08 cm cube of solidified waste is compressed vertically until failure. The strength at failure is recorded in kilopascals. One test consists of three replicates.

3.1.6 *Freeze/Thaw Weathering Resistance*

In cold climates, repeated cycles of freezing and thawing can cause physical deterioration of an exposed solidified waste product, thereby increasing contact with groundwater. The method for measuring freeze/thaw weathering resistance was adapted from the ASTM Test for Resistance of Concrete to

Rapid Freezing and Thawing (C666-80) and the ASTM Freezing and Thawing Tests of Compacted Soil-Cement Mixtures (D560-57), and has been standardized as ASTM Method D4843. A small cylindrical specimen of solidified waste is subjected to 12 cycles of freezing at -20°C for 24 hours, followed by thawing in water at room temperature for 24 hours. The weight loss of the specimen after each cycle is determined and compared with that of a control. The following features of the ASTM methods were retained: 1) freezing of the test specimens in air, followed by thawing in water; 2) the time periods and temperatures for each of the 12 cycles; and 3) the use of weight loss as a measure of freeze/thaw resistance. Modifications to the ASTM methods include 1) the use of smaller specimens, 2) adjustment of test specimen weight loss for control specimen weight loss, and 3) the lack of a mechanism to remove loose surface particles. The use of ultrasound to remove loose particles was studied during test development and found to be unnecessary. The time of the test was based on convenient working days, but implantation of a thermocouple in several specimens, during development of the test, showed that the specimens were completely frozen within 24 hours (Hannak et al., 1986).

3.1.7 Wet/Dry Weathering Resistance

Wetting and drying may also weather solidified wastes. The method used to measure wet/dry weathering resistance in this study has been standardized as ASTM Method D4842. A small cylindrical specimen is subjected to 12 cycles of drying at 60°C for 23 hours in a vacuum oven, followed by soaking in distilled water at room temperature for 23 hours. Again, the weight loss of the specimen is determined after each cycle and compared with that of a control. The specimen does not dry completely within the 23 hours in the vacuum oven, but a continuous flow of nitrogen through the oven is used to create standard, reproducible drying conditions (Hannak et al., 1986).

3.2 Chemical Evaluation Methods

The five chemical tests include three extractions, measurement of acid neutralization capacity, and a dynamic leaching test. The three extractions address the solubility and reactivity of contaminants when exposed to different reagents. The final concentration of the contaminant analyzed in the various extracts is limited by the achievement of equilibrium conditions. The dynamic leaching test examines diffusion-controlled leaching.

3.2.1 Equilibrium Extraction

This is a low liquid-to-solid ratio test designed for estimating the pore water composition of the solidified waste. A finely ground sample is mixed with distilled water at a 4:1 liquid-to-solid ratio until equilibrium is reached. A time period of 7 days was chosen from developmental tests (WTC, 1984).

3.2.2 U.S. EPA Toxicity Characteristic Leaching Procedure (TCLP)

This is a regulatory test designed to assist in waste classification (Method 1311). A sample of crushed waste is extracted with acetic acid solution for 18 hours at a 20:1 liquid-to-solid ratio. The chemical analysis of the extract is compared with a list of threshold contaminant levels to determine whether the waste exhibits hazardous characteristics. The method specifies the use of a zero headspace extractor to confine volatile organic contaminants. The zero headspace extractor was not used in this study, as no volatile contaminants were present in the wastes.

3.2.3 Acid Neutralization Capacity

The ability of a solidified waste to neutralize acid is important because the solubility of metal compounds depends upon pH. To determine the acid neutralization capacity, a ground sample is divided into 11 subsamples, which are placed in test tubes with an increased amount of nitric acid in each successive test tube. The liquid-to-solid ratio in each test tube is 4:1. Each tube is mixed for 48 hours to attain equilibrium before the extract pH is measured. The results may be expressed as a titration curve.

3.2.4 *Sequential Chemical Extraction*

This test was originally designed to determine metals speciation in sediments (Tessier et al., 1979; Fraser and Lum, 1983). An understanding of metals speciation in solidified wastes is helpful in determining the contaminants available for leaching in different chemical environments. A finely ground sample is treated by a series of five chemical extractions of increasing aggressiveness, ranging from ion exchange with lithium chloride to total digestion with hydrofluoric acid, to separate the contaminants into five fractions. The compounds in each successive fraction are more refractory than in the previous fraction.

3.2.5 *Dynamic Leach Test*

The dynamic leach test is the sole test investigated in this study that attempts to measure contaminant mobility. It is adapted from the American Nuclear Society Test ANSI/ANS 16.1 (ANS, 1986). A monolithic cylindrical specimen is immersed in distilled water at a volume to sample geometric surface area ratio of 10 cm. The leachant is renewed according to a schedule, with leaching intervals calculated using a diffusion model, such that the amount leached in each interval is equal (Côté and Isabel, 1984). Two schedules are available, for contaminants of greater or lesser mobility; the test may be performed for seven or nine leaching intervals, for total times of 5 or 9 days respectively. The fraction of the total amount present leached in each interval is used to calculate an apparent diffusion coefficient and a leachability index, which provide a measure of contaminant mobility. The method used differs from the American Nuclear Society method in that variable leaching intervals are used, and the overall test length is shorter.

3.3 *Chemical Analysis of Leachates*

The methods used to analyze contaminants in the leachates are summarized in Tables 9 and 10. Table 9 shows sample preparation procedures and Table 10 presents measurement methods.

3.4 *Micromorphological Evaluation Methods*

Studying the microscopic properties of solidified products provides insight into their macroscopic characteristics, both with respect to their gross engineering properties and with respect to their ability to immobilize contaminants. The methods used are described below.

3.4.1 *X-ray Diffraction*

X-ray diffraction (XRD) is used to identify the crystalline phases present in a sample. A finely ground sample is rotated while being irradiated with a monochromatic beam of X-rays. The different crystalline phases will diffract the beam of X-rays at different angles of rotation (θ) and produce strong signals that are recorded digitally, or on a strip chart recorder, as counts per second versus the 2θ angle. The peak intensities are related to the degree of sample crystallinity. The angles at which peaks occurred can be used with Bragg's law to calculate the spacing between the atomic planes of the different crystalline phases. A data base of known atomic plane spacings is used to determine the nature of these crystalline phases (JCPDS, 1986).

3.4.2 *Scanning Electron Microscopy*

Scanning electron microscopy (SEM) is used to create a magnified image of the solidified waste surface. A high energy focused beam of electrons, several tens of angstroms in diameter, scans a gold or carbon coated sample. Interaction of the beam with the outermost electronic shells of the atoms on the surface produces secondary electrons that are collected by a detector and displayed on a

Table 9 Sample Preparation Procedures

Waste	Contaminants of Interest	Extraction Methods
WTC synthetic solution	Arsenic	Sulphuric/nitric digestion[a]
	Cadmium	Aqua regia/H_2O_2 digestion[a,b]
	Chromium	Aqua regia/H_2O_2 digestion[b]
	Lead	Aqua regia/H_2O_2 digestion[b]
	Phenol	4AAP method[c]
WES synthetic sludge	Cadmium	Aqua regia/H_2O_2 digestion[b]
	Chromium	Aqua regia/H_2O_2 digestion[b]
	Nickel	Aqua regia/H_2O_2 digestion[b]
	Mercury	Cr_2O_7/nitric/cold vapour method[a]
Aluminum coil plating sludge	Arsenic	Sulphuric/nitric digestion
	Chromium	Aqua regia/H_2O_2 digestion[b]
	Lead	Aqua regia/H_2O_2 digestion[b]
	Thallium	Aqua regia/H_2O_2 digestion[b]
	Cyanide	Alkaline distillation[a]
Dredged sediment	Chromium	Aqua regia/H_2O_2 digestion[b]
	Copper	Aqua regia/H_2O_2 digestion[b]
	Lead	Aqua regia/H_2O_2 digestion[b]
	Zinc	Aqua regia/H_2O_2 digestion[b]
	Mercury	Cr_2O_7/nitric/cold vapour method
	Polychlorinated biphenyls (PCBs)	WTC GC method[a] or AEC GC method (PCB)[d] or U.S. EPA method 8080 (GC)[e]
Wood preservation waste soil	Arsenic	Sulphuric/nitric digestion
	Lead	Aqua regia/H_2O_2 digestion[b]
	Pentachlorophenol (PCP)	U.S. EPA method 8040 (GC)[e] or AEC GC method (PCP)[d]
	Polyaromatic hydrocarbons (PAHs)	AEC HPLC method (PAH)[d] or U.S. EPA method 610 (HPLC)[e]

[a] Source: WTC (1985).

[b] Digestion was carried out on TCLP leachates at the Wastewater Technology Centre and the Alberta Environmental Centre only; metals in other leachates, or analyzed by the Waterways Experiment Station, were measured directly.

[c] Source: APHA (1981).

[d] Source: AEC (1985b).

[e] Source: U.S. EPA (1980).

cathode ray tube screen, resulting in an image of the surface. Magnifications ranging from 20 to 10 000 times the original size can be achieved (Goldstein et al., 1984).

3.4.3 Energy Dispersive X-ray Analysis

Energy dispersive X-ray (EDXR) analysis can be used with scanning electron microscopy to determine the local composition of crystalline grains. An analysis can be obtained from a volume of 1 μm^3; thus, energy dispersive X-ray analysis can provide information about the distribution of elements in a sample, which cannot be determined using bulk chemical methods. In addition to producing electrons, the interaction of the high energy electron beam with the surface atoms also produces characteristic X-rays of different energies. The relative quantities of the elements present in a sample volume are determined from the intensity and energy of the X-rays. The detection limit of the method is dependent upon the atomic number of the element of interest, and is approximately 0.5 wt.%.

3.4.4 Optical Microscopy

Optical microscopy provides information about the gross distribution of different phases. The size of the crystals in solidified

Table 10 Measurement Methods

Parameter	Instrument Used in Laboratory		
	WTC	AEC	WES
Arsenic	Hyd. gen./ICP	GFAA	GFAA
Mercury	AAS	GFAA	AAS
Other metals	DCP and GFAA	ICP	GFAA
Cyanide	Colorimetry	Colorimetry	Colorimetry
Total organic carbon (TOC)	DC80	Colorimetry	Carbon analyzer
Phenol	Colorimetry	Colorimetry	NA
Polychlorinated biphenyls (PCBs)	GC (ECD)	GC (ECD)	GC (ECD)
Pentachlorophenol	GC (ECD)	GC (ECD)	GC/MS
Polyaromatic hydrocarbons (PAHs)	HPLC	HPLC	GC/MS

Note: Hyd. gen., hydride generator.
ICP, inductively coupled plasma spectrometry.
AAS, atomic adsorption spectrometry.
DCP, directly coupled plasma spectrometry.
GFAA, graphite furnace atomic adsorption spectrometry.
GC, gas chromatography.
MS, mass spectrometry.
ECD, electron capture detector.
HPLC, high performance liquid chromatography.
NA, not applicable.
DC80, Dohrmann Carbon Analyzer, Model DC80.

samples is likely to be close to the resolution of the microscope, but any large-scale sample heterogeneity should be apparent. In addition, the optical microscope allows the observation of colour. The solidified products were prepared in two ways for observation under an ordinary transmitted-light microscope: 1) grains of powdered sample were solidified in epoxy resin, which was ground to a thickness such that light was transmitted by most of the particles, and 2) powdered sample was sprinkled on glass slides with a few drops of water. The samples were observed at magnifications of 40 to 400 times the original size.

Methods for Analyzing Reproducibility

4.1 Statistical Design of Testing

The various solidified product specimens were distributed by Dynamac Corporation to the laboratories for physical and chemical testing according to the basic testing design shown in Table 11. According to this design, each solidified product was to be tested in duplicate in two laboratories, resulting in a total of four replicates of each test. Because of limited laboratory capacities, the basic design was modified for selected tests and wastes. Also, occasional errors or missing samples resulted in modifications to the basic design. Design modifications are summarized in the following paragraphs.

As originally planned, for each of the bulk density, specific gravity (wet or dry), water content, and unconfined compressive strength methods, a single test consisted of three within-test replicates. Consequently, conducting the basic design would result in a total of six replicates within each laboratory (two test replicates × three within-test replicates). In general, this was the case for the bulk density and unconfined compressive strength measurements for the solidified products of all the raw waste types. However, the water content measurement and the wet specific gravity method were carried out singly in each laboratory (i.e., three within-test replicates). Two replicates of the dry specific gravity method were carried out for most solidified products at the Wastewater Technology Centre (six total replicates), three replicates were conducted at the Alberta Environmental Centre (nine total replicates),

Table 11 **System of Sample Distribution (basic design, per waste, per test)**

Industrial Participant	Number of Samples to		
	WTC	AEC	WES
A	2	2	0
B	0	2	2
C	2	0	2
D	2	2	0
E	0	2	2
F	2	0	2
G	2	2	0
H	0	2	2
I	2	0	2
J	2	2	0
K	0	2	2
L	2	0	2
M	2	2	0
N	0	2	2
O	2	0	2

and none were conducted at the Waterways Experiment Station. For statistical purposes, it was most practical to deal with all replicates of these tests in each of the laboratories together, rather than separating them into groups of three.

The basic testing design was applied for hydraulic conductivity measurement of the Waterways Experiment Station synthetic sludge specimens, the aluminum coil plating sludge specimens, and the wood preservation waste soil specimens.

The weathering tests were performed according to the basic design for all solidified products, with the exception of the solidified dredged sediment, since it is unlikely that the sediment would be exposed to weathering conditions.

The equilibrium extraction test and the toxicity characteristic leaching procedure were applied to the number of samples specified in the basic design for all the solidified products.

The acid neutralization capacity was not measured for the dredged sediment solidified products or the wood preservation waste soil products, since they did not contain large quantities of heavy metals. This test was also not applied to products of the aluminum coil plating sludge as they contained cyanide that would have been released by the addition of acid.

The sequential chemical extraction test and the dynamic leach test were not carried out at all by the Waterways Experiment Station. The sequential chemical extraction test was conducted in duplicate by the Wastewater Technology Centre and the Alberta Environmental Centre for solidified products of the wastes containing metals, i.e., the Wastewater Technology Centre synthetic solution, the Waterways Experiment Station synthetic sludge, the aluminum coil plating sludge, and the dredged sediment. The dynamic leach test was conducted singly by the Wastewater Technology Centre and the Alberta Environmental Centre for the Wastewater Technology Centre synthetic solution products, the aluminum coil plating sludge products, and the wood preservation waste soil products.

The micromorphological characterization techniques were applied by Louisiana State University to all solidified products of the Wastewater Technology Centre synthetic solution, the Waterways Experiment Station synthetic sludge, and the wood preservation waste soil. Depending upon the individual sample characteristics, analysis by one of the techniques may have been repeated at different magnifications or omitted entirely.

4.2 Quality Control

Standard samples, blank samples, split samples, and split blank samples were submitted to each laboratory for analytical quality control. Duplicate standard samples containing known concentrations of the contaminants of interest were prepared at the Wastewater Technology Centre and sent to the Alberta Environmental Centre and the Waterways Experiment Station for testing. Blank samples were run in each laboratory with each batch of equilibrium extractions and toxicity characteristic leaching procedures. In addition, each of the three laboratories divided an equilibrium extract and a toxicity characteristic leaching procedure leachate, and the corresponding blanks, for one solidified product of each raw waste type, for analysis in all three laboratories.

The amount of quality control data (for standards, blanks, and splits) was insufficient to allow firm conclusions regarding the validity of the remaining analytical data to be drawn. Statistical treatment of the data from each test, as discussed in the following sections, was more helpful.

4.3 *Statistical Analysis of Results*

In determining the suitability of a test method for characterizing solidified wastes, the reproducibility of the method within and between laboratories must be assessed. The statistical techniques used to analyze the reproducibility of the tests are outlined in the following sections. The referenced texts provide more detail regarding these techniques.

Statistical analysis was performed on comparable data only, i.e., if any data for a sample were missing, incomplete, or below detection, that sample was omitted from the analysis. The number of degrees of freedom is a function of the number of data points used in a calculation.

4.3.1 Intralaboratory Reproducibility

To measure each parameter within each laboratory, an average value ($\bar{y}$) and a variance (s^2) were determined for each solidified product according to the following equations:

$$\bar{y} = \frac{1}{n} \sum_{i=1}^{n} y_i \qquad (1)$$

$$s^2 = \frac{1}{(n-1)} \sum_{i=1}^{n} (y_i - \bar{y})^2 \qquad (2)$$

where n is the number of replicates and y_i is the ith replicate.

It was assumed that the variance was solidified product independent and that, for each parameter, the variances for the different solidified products could be pooled as follows (Box et al., 1978):

$$s^2 = (\sum_{i=1}^{n} \upsilon_i \; s_i^2) / \sum_{i=1}^{n} \upsilon_i \qquad (3)$$

where v_i is the number of degrees of freedom for measurements for the ith solidified product. The intralaboratory variances for each of the three laboratories were averaged as well, to estimate an overall-pooled intralaboratory variance.

This approximate overall-pooled intralaboratory variance was used to estimate the number of replicates of each test required to attain a desired precision in the parameter (Reckhow and Chapra, 1983):

$$n = \frac{t^2_{0.025} \, s^2}{d^2} \qquad (4)$$

where $t_{0.025}$ is Student's t statistic corresponding to the area in both tails of the t distribution, such that $\Pr(|t| > t_{0.025}) = 0.05$, and the precision, d, is defined as the difference between the population mean (i.e., the true value of the measurement) and the sample mean (i.e., the average of n replicates). This equation may be rearranged to determine the level of precision attained by using a given number of replicates.

To apply the above equations, the variance must be independent of the value of the parameter. For bulk density, specific gravity (by both methods), water content, and freeze/thaw and wet/dry weathering resistance, this required independence was apparent. For the hydraulic conductivity, the unconfined compressive strength, and the concentration data from the leaching tests, the standard deviation (s, the square root of the variance) appeared to be proportional to the value of the parameter. It is not unusual for a population distribution to be lognormal, particularly for quantitative analytical chemistry over a wide range of concentrations. Thus, for these parameters the variance stabilizing transformation $x = \log(y)$ was applied (Sachs, 1984). Equation 1 was applied to the transformed data (i.e., x), resulting in geometric rather than arithmetic averages. The transformed data were also manipulated using Equations

1, 2, and 3. To apply Equation 4 to the transformed data, the precision, d, was redefined as the logarithm of the ratio of the population mean to the sample mean.

4.3.2 Interlaboratory Reproducibility

Using the average value of each parameter measured in each laboratory for each solidified product, interlaboratory averages and variances were calculated for each of the three laboratory pairs. An interlaboratory variance could not be simply calculated for all three laboratories, since testing for each solidified product was conducted in only two of the three laboratories. As was done for the intralaboratory variances, the interlaboratory variances were pooled across the solidified products, and across the interlaboratory pairs to determine an overall-pooled interlaboratory variance. An F test (Box et al., 1978) was conducted to compare the interlaboratory and intralaboratory variances.

The overall-pooled interlaboratory variance can be used to determine the width of the 95% confidence interval on a parameter for each of the solidified products: 2 × $t_{0.025}$ × s, where $t_{0.025}$ is as defined earlier, and the standard deviation, s, is the square root of the overall-pooled interlaboratory variance. For eight or more degrees of freedom, $t_{0.025}$ is approximately equal to 2; therefore, the width of the 95% confidence interval is approximately four times the standard deviation.

If the 95% confidence interval for the measurement for each individual solidified product is as wide (or wider) than the range of values encountered across the different solidified products, this indicates that the spread of data for the different solidified products could have been caused by experimental error only. However, if the observed range is, in fact, greater than might be expected from the experimental error (as indicated by the 95% confidence interval), it may be concluded that a real difference between solidified products has been measured.

To allow comparison of the 95% confidence interval on a value for one solidified product, with the range of values measured for the different solidified products, a 95% confidence interval was calculated for the median value of the parameter, m, according to the following formula:

$$95\%\ \textit{confidence interval} = m \pm t_{0.025} \times s \qquad (5)$$

Since t becomes large when the number of degrees of freedom is small, the confidence interval becomes very wide if a small data set is used to calculate the variance. Therefore, a confidence interval was not calculated if there were fewer than eight degrees of freedom, since the width of the confidence interval would reflect the lack of data rather than the sample variability.

For the data that were logarithmically transformed, the 95% confidence interval for the median parameter was calculated from the logarithmic overall-pooled interlaboratory standard deviation, s_x, as follows:

$$95\%\ \textit{confidence interval} = m \times/ \textit{antilog}(t_{0.025} \times s_x) \qquad (6)$$

where ×/ means to multiply or divide. This confidence interval may also be described as ± a percentage, as has been done throughout this report.

In addition, for each solidified product, the average value of a parameter measured in one laboratory (a) was plotted against the average value measured in the other testing laboratory (b). Ideally, the points for each laboratory pair should fall on the line with the equation a = b. A correlation coefficient, r, was calculated to determine the degree of scatter from a linear relationship between the values determined in the two laboratories:

$$r^2 = \left(\sum_{i=1}^{n} a_i b_i\right) / \left(\sum_{i=1}^{n} a_i^2 \sum_{i=1}^{n} b_i^2\right) \qquad (7)$$

An r value of one indicates a perfect linear relationship between the two sets of data, whereas no relationship exists if r is zero. In this report, an r value greater than 0.8 was considered to show a good relationship between the two sets of data, and some degree of relationship was presumed to exist for r greater than 0.6. If r was less than 0.6, it was concluded that no definite relationship between the two variables existed. Unfortunately, however, the significance of the correlation coefficient diminishes with the number of points used in calculating it. Although a correlation coefficient was calculated if more than two comparable points existed, too much importance should not be placed upon its value.

Since a correlation coefficient measures only whether a relationship exists between the values determined in the two laboratories, and not whether there is a bias in either laboratory, and a least-squares linear regression is inappropriate when there is error in both variables, a paired t-test was also carried out to compare the average difference between the laboratories with a null hypothesis of a zero difference.

Section 5

Test Method Results and Reproducibility

The following sections summarize the results measured for each property and the statistical analysis of these results, which was carried out as described in Section 4. The reproducibility of each test method within and between laboratories is considered in an absolute sense, and also relative to the range of properties that may be encountered and the intended use of the measurement.

5.1 Physical Evaluation Results

For each of the physical test methods applied to the solidified products, an average value was calculated for each solidified product, as described in Section 4. Tables in the following sections summarize the interlaboratory averages as medians and ranges of the parameter values, across the different solidified products. For each property, the overall-pooled intralaboratory standard deviation, the overall-pooled interlaboratory standard deviation, and the 95% confidence interval on the median of the given range (based on the interlaboratory standard deviation) are also presented. For each test, the recommended number of replicates was calculated based on the overall-pooled intralaboratory reproducibility. These are summarized in Section 7.

5.1.1 Bulk Density

Table 12 shows the median and range for the bulk densities of the solidified products from each waste type, and summarizes the statistical analysis. The ranges were determined by averaging bulk densities for each solidified product across the laboratories. For the solidified products of all five wastes, the median bulk density was 1.44 g/cm^3, with bulk densities ranging from less dense than water, at 0.80 g/cm^3, to 2.18 g/cm^3. The ranges for the solidified products of all the waste types were similar, but the bulk densities for the wood preservation waste soil solidified products were slightly higher because the soil was initially dry.

The overall-pooled interlaboratory variance was used to calculate a 95% confidence interval on the median bulk density measured in this study: 1.44 ± 0.21 g/cm^3. The range of bulk densities encountered for the different solidified products exceeds the interlaboratory 95% confidence interval on the median bulk density. Thus, a real difference in bulk densities was measured for the different solidified products. Since the materials used in the various solidification processes were apparently very similar, the observed range primarily represents differences in porosity.

An F test was used to compare the overall-pooled interlaboratory variance with the overall-pooled intralaboratory variance; the interlaboratory variance was greater than the intralaboratory variance at the 1% significance level.

Inspection of the laboratory pair plots of the average measured bulk densities for each solidified product in each laboratory shows that the data points approximate a line with the equation x = y. The correlation coefficients for these data points are greater than 0.8, indicating little scattering from a straight line and a good relationship between the results from the different laboratories. A t-test comparison of the average difference between the laboratory pairs with a hypothetical difference of zero shows that, at the 1% level, no significant difference exists between average values measured at the Wastewater Technology Centre and the Alberta Environmental Centre, or between values measured at the Wastewater

Table 12 **Summary of Bulk Densities for All Solidified Products**

Raw Waste	Number of Products Tested	Average Bulk Density (g/cm^3) Median	Range
WTC synthetic solution	14	1.43	0.91 to 1.87
WES synthetic sludge	14	1.42	0.80 to 1.69
Aluminum coil plating sludge	13	1.34	0.83 to 1.99
Dredged sediment	15	1.40	0.99 to 1.97
Wood preservation waste soil	13	1.57	1.17 to 2.18
Overall	69	1.44	0.80 to 2.18

Summary of Statistics	Degrees of Freedom	Standard Deviation	95% C.I. for Median
Intralaboratory*	472	0.057	-
Interlaboratory**	60	0.105	1.23 to 1.65

Note: C.I., confidence interval.

* For a single bulk density measurement.

** For a group of three bulk density measurements in each laboratory.

Technology Centre and the Waterways Experiment Station. However, bulk densities measured at the Waterways Experiment Station are significantly higher, by 0.08 g/cm^3, than bulk densities measured at the Alberta Environmental Centre. Such a small difference is of no practical importance.

5.1.2 Specific Gravity

The specific gravity of the solidified products was measured using two methods. The results of the more traditional method of using water to fill the pores of the matrix were compared with a method using helium to fill the pores, since it was suspected that the use of water resulted in partial dissolution of the sample matrix.

The medians and ranges of specific gravity results for the wet method, for all five waste types, and a summary of the statistical analysis are presented in Table 13. For all samples tested, the median specific gravity measured using the wet method was 2.48, with a range from 1.75 to 3.53. The ranges were similar for the solidified products of all five waste types.

The 95% confidence interval on the median specific gravity for all the solidified products, calculated using the overall-pooled interlaboratory variance, was 2.48 ± 0.66. Since the observed range of specific gravities only slightly exceeded the range described by the interlaboratory 95% confidence interval, it appears that there is a difficulty in measuring real differences between solidified products with this method.

Comparison of the overall intralaboratory and interlaboratory variances using an F test at the 1% significance level showed that the interlaboratory variance was significantly greater than the intralaboratory variance.

As might be expected, the laboratory pair plots for the average specific gravities measured using the wet method indicate no apparent relationship between the value measured in one laboratory and that measured in another. The low correlation coefficients calculated for the data confirm this lack of relationship. However, the t-test comparisons of means for each of the laboratory pairs show that there is no bias in the results from

Table 13 **Summary of Specific Gravities Measured Using the Wet Method for All Solidified Products**

Raw Waste	Number of Products Tested	Average Specific Gravity Median	Range
WTC synthetic solution	14	2.55	2.27 to 3.53
WES synthetic sludge	14	2.45	1.75 to 2.80
Aluminum coil plating sludge	13	2.48	2.21 to 2.80
Dredged sediment	15	2.53	2.39 to 2.92
Wood preservation waste soil	13	2.29	2.11 to 2.70
Overall	69	2.48	1.75 to 3.53

Summary of Statistics	Degrees of Freedom	Standard Deviation	95% C.I. for Median
Intralaboratory*	242	0.17	-
Interlaboratory**	66	0.33	1.82 to 3.14

Note: C.I., confidence interval.

* For a single specific gravity measurement.

** For a group of three specific gravity measurements in each laboratory.

any of the three different laboratories at the 1% level.

For the dry method, the median specific gravity for all the solidified products was 2.34, with values ranging from 1.94 to 2.71. The data are presented in Table 14 for each waste, with a summary of the statistical analysis. Specific gravities were measured using the dry method at the Wastewater Technology Centre and the Alberta Environmental Centre only.

Using the overall-pooled interlaboratory variance, the 95% confidence interval was 2.34 ± 0.20. Again, for the dry method, the range of measured values does not greatly exceed the interlaboratory 95% confidence interval, showing the inability of the test to differentiate easily between solidified products.

An F-test comparison of the pooled interlaboratory variance for the Wastewater Technology Centre and the Alberta Environmental Centre with the overall-pooled intralaboratory variance indicated a much greater variability between laboratories than within laboratories at the 1% significance level.

The laboratory pair plot for the average specific gravities measured using the dry method at the Wastewater Technology Centre versus those measured at the Alberta Environmental Centre shows some scattering from a linear relationship. The correlation coefficient for the data is 0.7, confirming a relationship between the results obtained in the two laboratories. A t-test comparison of the specific gravities in the two laboratories shows that specific gravities measured at the Alberta Environmental Centre tend to be higher than those measured at the Wastewater Technology Centre by an average of 0.07 at the 1% significance level.

Comparing the results from the two methods, we observe that the variability associated with the dry method is lower than that associated with the wet method; correspondingly, the observed range of values from the dry method

Table 14 **Summary of Specific Gravities Measured Using the Dry Method for All Solidified Products**

Raw Waste	Number of Products Tested	Average Specific Gravity	
		Median	Range
WTC synthetic solution	9	2.27	2.06 to 2.17
WES synthetic sludge	8	2.37	1.94 to 2.59
Aluminum coil plating sludge	12	2.35	2.16 to 2.47
Dredged sediment	13	2.51	2.23 to 2.67
Wood preservation waste soil	13	2.31	2.17 to 2.43
Overall	55	2.34	1.94 to 2.71

Summary of Statistics	Degrees of Freedom	Standard Deviation	95% C.I. for Median
Intralaboratory*	495	0.023	-
Interlaboratory**	17	0.1	2.14 to 2.54

Note: C.I., confidence interval.

* For a single specific gravity measurement.

** For a group of three specific gravity measurements in each laboratory.

is also narrower (since the values include less experimental error). The correlation coefficient between the two methods was 0.6, showing a weak relationship between the two methods. Comparison of the two methods using a t-test at the 1% significance level showed that the wet method tended to result in specific gravities that were higher by an average of 0.13. Such a difference could be attributable to partial dissolution of the sample matrix in water.

The inability of either method to easily distinguish between different solidified products may be a consequence of the similarity of the solidification ingredients used; it is clear that cement-based solidified products should have specific gravities in the neighbourhood of the specific gravity of cement. The specific gravity of hydrated portland cement is approximately 2.5 (Brunauer, 1957), whereas the median specific gravity measured using the wet method is 2.5 and the median specific gravity measured using the dry method is 2.3.

5.1.3 Moisture Content

The moisture content ranges and medians for the solidified products of each raw waste type, and the summarized statistical data, are listed in Table 15. Across the various solidified products, moisture contents ranged from almost negligible, at 0.88% (w/ww), to 63.6%, with a median of 25.8%. The moisture content ranges for the solidified products for all the waste types were similar, with the exception of the wood preservation waste soil solidified products, which appeared to have lower moisture contents.

A 95% confidence interval for the median moisture content was also determined using the overall-pooled interlaboratory variance: 25.8 ± 10.2% (w/ww). The range of moisture contents measured is much wider than the interlaboratory 95% confidence interval, indicating clearly that real differences in moisture contents exist for the different solidified products.

An F test showed that the overall interlaboratory variance was much higher

Table 15 Summary of Moisture Contents for All Solidified Products

Raw Waste	Number of Products Tested	Average Moisture Content (%, w/ww) Median	Range
WTC synthetic solution	14	19.9	1.1 to 63.6
WES synthetic sludge	14	26.0	2.4 to 45.7
Aluminum coil plating sludge	13	33.6	10.3 to 54.0
Dredged sediment	15	29.7	0.93 to 48.0
Wood preservation waste soil	13	12.6	0.88 to 24.8
Overall	69	25.8	0.88 to 63.6

Summary of Statistics	Degrees of Freedom	Standard Deviation	95% C.I. for Median
Intralaboratory*	260	0.65	-
Interlaboratory**	63	5.1	15.6 to 36.0

Note: C.I., confidence interval.

* For a single moisture content measurement.

** For a group of three moisture content measurements in each laboratory.

than the intralaboratory variance at the 1% significance level. Difficulty in standardizing drying oven conditions, and variations in laboratory humidity in different geographical locations may explain the observed difference.

Inspection of the laboratory pair plots of the average moisture contents measured in each laboratory shows that the data points approximate a line with the equation $x = y$, with some scattering. The correlation coefficients for these laboratory pair data are 0.8 or greater, which also indicates a strong relationship between the results found in the different laboratories. According to the t-test, there is no consistent bias in the results from any of the three laboratories.

5.1.4 Calculated Physical Properties

The weight change factors calculated using the information provided by the industrial participants concerning their processes (see Section 2.2) were used with bulk density data to calculate a volume change factor for each solidification process. The average bulk density, specific gravity, and moisture content were used to calculate the porosity and degree of saturation for each solidified product. Wherever possible, the specific gravity measured using the dry method was used to calculate the porosity and degree of saturation, since it was previously shown to be more accurate than that measured by the wet method. The ranges for the volume change factor and porosity are summarized in Tables 16 and 17 respectively. The power law for the propagation of errors was used to calculate a standard deviation for the median value of each parameter from the overall-pooled interlaboratory variances associated with the bulk density, specific gravity, and moisture content.

The volume change factor (VCF) for a solidification process was calculated using the following formula:

$$VCF = \frac{BD_r}{BD} \times WCF \qquad (8)$$

where BD_r = bulk density of the raw waste (g/cm^3)

Table 16 **Summary of Volume Change Factors for All Solidified Products**

Raw Waste	Number of Products	Volume Change Factor	
		Median	Range
WTC synthetic solution	14	1.70	1.01 to 3.67
WES synthetic sludge	14	1.67	1.23 to 3.73
Aluminum coil plating sludge	13	1.64	1.12 to 3.95
Dredged sediment	15	1.41	1.14 to 2.84
Wood preservation waste soil	13	1.36	0.96 to 4.04
Overall	69	1.55	0.96 to 4.04
Summary of Statistics		Standard Deviation	95% C.I. for Median
Interlaboratory		0.15	1.25 to 1.85

Note: C.I., confidence interval.

Table 17 **Summary of Calculated Porosities for All Solidified Products**

Raw Waste	Number of Products	Porosity	
		Median	Range
WTC synthetic solution	14	0.55	0.21 to 0.81
WES synthetic sludge	14	0.56	0.32 to 0.74
Aluminum coil plating sludge	13	0.64	0.27 to 0.76
Dredged sediment	15	0.59	0.23 to 0.74
Wood preservation waste soil	13	0.44	0.16 to 0.50
Overall	69	0.57	0.16 to 0.81
Summary of Statistics		Standard Deviation	95% C.I. for Median
Interlaboratory		0.095	0.38 to 0.76

Note: C.I., confidence interval.

BD = bulk density of the solidified product (g/cm^3)

WCF = weight change factor for the solidification process

The volume change factors for the different solidification processes ranged from 0.96, representing a slight reduction in the volume of the solidified product with respect to the raw waste, to 4.04, a fourfold increase in volume, with a median of 1.55. Using the variance associated with the bulk density measurement, a 95% confidence interval was calculated for the median volume change factor: 1.55 ± 0.30. The variance associated with the weight change factor was assumed to be negligible.

The range of calculated volume change factors substantially exceeded the interlaboratory 95% confidence interval on the median volume change factor, demonstrating that the solidified products were measurably very different from one another in this respect.

The porosity (P) of the solidified waste matrices was calculated using the following equation:

$$P = 1 - \frac{BD\,(1 - W)}{SG \times d} \qquad (9)$$

where W = moisture content of the solidified product (w/ww)

SG = specific gravity of the solidified product

d = density of water (1 g/cm^3)

It was found that, for all the solidified products, the porosity ranged from 0.16 to 0.81, with a median porosity of 0.57. The 95% confidence interval calculated for this median porosity was 0.57 ± 0.19.

The calculated range of porosities only slightly exceeded the interlaboratory 95% confidence interval on the median porosity, indicating the difficulty in measuring differences in porosity. The error associated with the porosity numbers was relatively high because of the combined effect of the errors associated with the measured properties used in the calculation.

5.1.5 Hydraulic Conductivity

Table 18 shows the median and range for the hydraulic conductivities of the solidified products from each waste type, and summarizes the statistical analysis. For the solidified products of all three wastes, the median hydraulic conductivity was 3×10^{-8} m/s, with hydraulic conductivities ranging from that of a dense clay, at 7×10^{-10} m/s, to that of silty sand, at 3×10^{-6} m/s. The hydraulic conductivity appears to be independent of the raw waste type. Since the standard deviation appeared

Table 18 Summary of Hydraulic Conductivities for All Solidified Products

Raw Waste	Number of Products Tested	Average Hydraulic Conductivity (m/s) Median	Average Hydraulic Conductivity (m/s) Range
WES synthetic sludge	12	6×10^{-9}	2×10^{-9} to 3×10^{-6}
Aluminum coil plating sludge	13	3×10^{-8}	7×10^{-10} to 2×10^{-6}
Wood preservation waste soil	12	5×10^{-8}	2×10^{-9} to 2×10^{-6}
Overall	37	3×10^{-8}	7×10^{-10} to 3×10^{-6}

Summary of Statistics	Degrees of Freedom	Percent Standard Deviation	95% C.I. for Median
Intralaboratory: Between run*	111	32	-
Between test**	52	249	-
Interlaboratory**	25	312	2×10^{-9} to 5×10^{-7}

Note: C.I., confidence interval.

* For hydraulic conductivity runs on the same sample.

** For hydraulic conductivity tests on different samples.

to be proportional to the magnitude of the hydraulic conductivity, statistics were performed on the logarithmic transformations of the data. The ranges were determined by calculating the geometric average hydraulic conductivity for each solidified product across the laboratories.

The overall-pooled interlaboratory variance was used to calculate a 95% confidence interval: 3×10^{-8} ± 1600% (i.e., ×/ 17) m/s. The range of measured hydraulic conductivities somewhat exceeds the interlaboratory 95% confidence interval on the median, but it appears that the four orders of magnitude spread in the measured hydraulic conductivities is largely the result of experimental error.

In each of the three laboratories, several runs were carried out for each sample. Within the Wastewater Technology Centre and the Waterways Experiment Station, replicates of the hydraulic conductivity measurement were also performed on two specimens of each solidified product. As a result, intralaboratory variances were calculated for replicate hydraulic conductivity measurements on the same specimen (runs) and on different specimens (tests). The overall-pooled intralaboratory between-test variance was used to calculate the recommended number of replicates for this test.

An F test was used to compare the overall-pooled intralaboratory between-run variance with the overall-pooled intralaboratory between-test variance, and the overall-pooled interlaboratory variance with the overall-pooled intralaboratory between-test variance. The between-test variance was much greater than the between-run variance at the 1% significance level. Thus, it appears that the difference between individual specimens of the same solidified product is the greatest source of variability with respect to the hydraulic conductivity measurement. The interlaboratory variance was not higher than the intralaboratory between-test variance at the 1% significance level.

The laboratory pair plots of the average hydraulic conductivity measured in each laboratory are scattered in appearance. The correspondence between data measured at the Wastewater Technology Centre and the Alberta Environmental Centre is the worst, with a correlation coefficient of only 0.27; the correlation coefficient between data from the Waterways Experiment Station and the Alberta Environmental Centre is 0.54, and that for the Wastewater Technology Centre and the Waterways Experiment Station is fairly good, at 0.8. A t-test shows that, at the 1% level, no significant difference exists between average values measured at the Wastewater Technology Centre, the Alberta Environmental Centre, or the Waterways Experiment Station.

5.1.6 Unconfined Compressive Strength

The median unconfined compressive strength for all the solidified products tested was 1760 kPa, with values ranging from 70 to 17 880 kPa. The values encountered for the solidified products of each raw waste type are given in Table 19, along with the summarized statistical data. As was the case for the hydraulic conductivity data, the standard deviation appeared to be a function of the magnitude of the unconfined compressive strength, so the statistical analysis was performed on the logarithmic transformations of the data.

Comparison of the overall intralaboratory and interlaboratory variances using an F test at the 1% significance level showed that the interlaboratory variance was greater.

A 95% confidence interval was calculated using the overall-pooled interlaboratory variance: 1760 ± 171% (i.e., ×/ 2.71) kPa. The range of measured strengths was much wider than the range described by the interlaboratory 95% confidence interval, showing that real differences between solidified products were measured.

Table 19 Summary of Unconfined Compressive Strengths for All Solidified Products

Raw Waste	Number of Products Tested	Average Unconfined Compressive Strength (kPa) Median	Range
WTC synthetic solution	12	3 900	260 to 16 600
WES synthetic sludge	13	1 370	70 to 17 580
Aluminum coil plating sludge	13	990	130 to 17 880
Dredged sediment	15	910	90 to 17 630
Wood preservation waste soil	13	4 310	120 to 14 780
Overall	66	1 760	70 to 17 880

Summary of Statistics	Degrees of Freedom	Percent Standard Deviation	95% C.I. for Median
Intralaboratory*	441	32	-
Interlaboratory**	59	65	650 to 4 780

Note: C.I., confidence interval.

* For a single unconfined compressive strength measurement.

** For a group of three unconfined compressive strength measurements in each laboratory.

Inspection of the laboratory pair plots for the average logarithmic unconfined compressive strength data in each laboratory shows a clear relationship between the values measured in the different laboratories, which is confirmed by correlation coefficients near 0.9 for all the data. A t-test comparison of the average difference between the laboratory pairs with a hypothetical difference of zero shows no difference between the results measured in the different laboratories at the 1% significance level.

5.1.7 Freeze/Thaw Weathering Resistance

The results of this test allow the samples to be divided into two categories: 1) those that failed before the end of 12 freeze/thaw cycles (where failure is defined as greater than 30% cumulative corrected relative weight loss), and 2) those that survived the 12 cycles. It was found that 56% of the solidified products did not survive all 12 cycles of the test. For these, the ranges of the number of cycles to failure for the solidified products of each waste type are reported in Table 20a. The number of cycles to failure ranged from one to more than 12,* with a median of 7. For the solidified products that survived 12 cycles, the ranges for the cumulative corrected relative weight loss at the end of 12 cycles for the solidified products of each waste type are given in Table 20b. A negative weight loss is caused by greater control specimen weight loss than test specimen weight loss. Overall, the cumulative corrected relative weight loss at the end of 12 cycles ranged from -0.17 to

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* For some solidified products, some replicates failed before the end of 12 cycles, whereas other replicates survived more than 12 cycles. If this was the case, the solidified product was included with others that did not survive 12 cycles. The number of cycles to failure for the replicates that survived 12 cycles was assumed to be 13 for the purpose of calculating averages.

Table 20 Summary of Freeze/Thaw Test Results

(a) Solidified products that did not survive 12 cycles

Raw Waste	Number of Products Tested	Number Not Surviving 12 Cycles	Number of Cycles to Failure	
			Median	Range
WTC synthetic solution	12	6	8	1 to 9.5
WES synthetic sludge	14	10	6.4	1 to 12.3
Aluminum coil plating sludge	13	9	6.5	1.5 to 11.8
Wood preservation waste soil	13	4	4.4	1.5 to 12.8
Overall	52	29	7	1 to 12.8

Summary of Statistics	Standard Deviation	95% C.I. for Median
Intralaboratory	1.6	-
Interlaboratory	2.0	3 to 11

Note: C.I., confidence interval.

(b) Solidified products that survived 12 cycles

Raw Waste	Number Surviving 12 Cycles	Weight Loss After 12 Cycles (%, w/dw)	
		Median	Range
WTC synthetic solution	6	0.01	-0.035 to 4.6
WES synthetic sludge	4	4.4	0.61 to 8.1
Aluminum coil plating sludge	4	4.0	0.97 to 7.0
Wood preservation waste soil	9	0.92	-0.17 to 5.5
Overall	23	0.97	-0.17 to 8.1

Summary of Statistics	Standard Deviation	95% C.I. for Median
Intralaboratory	2.6	-
Interlaboratory	2.2	-3.4 to 5.3

Note: A negative weight loss indicates greater control specimen weight loss than test specimen weight loss.
C.I., confidence interval.

8.1% (w/dw), with a median of 0.97%. From these ranges for the number of cycles to failure, and the weight loss after 12 cycles, it can be seen that specimens that survived the 12 cycles tended to survive them with minimal weight loss.

The overall-pooled interlaboratory variances were used to calculate 95% confidence intervals for the median number of cycles to failure (7 ± 4) and the median cumulative corrected relative weight loss after 12 cycles (0.97 ± 4.4%). For the number of cycles to failure, the observed range exceeds the interlaboratory 95% confidence interval on the median number of cycles to failure, showing that differences between solidified products can be, and were, measured. For the weight loss at 12 cycles, the observed range and the 95% confidence interval on the median are very similar, suggesting that there is no measurable difference between these more durable products, at least up to 12 cycles.

Comparison of the overall intralaboratory and interlaboratory variances using an F test at the 1% significance level showed that, for the number of cycles to failure, the interlaboratory variance was greater, but for the weight loss measured after 12 cycles, there was no significant difference between the variability within and between laboratories.

The laboratory pair plots for the number of cycles to failure are somewhat scattered, with correlation coefficients between the data ranging from 0.5 to 0.6. For the cumulative corrected relative weight loss after 12 cycles, the correlation between values measured at the Wastewater Technology Centre and the Alberta Environmental Centre is poor, with a correlation coefficient of 0.04. However, between the Wastewater Technology Centre and the Waterways Experiment Station, and the Alberta Environmental Centre and the Waterways Experiment Station, the correlation between weight loss data is good, with correlation coefficients of 0.9 and 0.7, respectively, which are reflected in the laboratory pair plots. The t-test at the 1% significance level shows no significant bias in any of the laboratories for either the number of cycles to failure or the weight loss after 12 cycles.

5.1.8 Wet/Dry Weathering Resistance

As was the case for the results from the freeze/thaw weathering test, the results of the wet/dry weathering test allow the samples to be divided into two categories: 1) those that failed before the end of the 12 wet/dry cycles (where failure is defined as greater than 30% cumulative corrected relative weight loss), and 2) those that survived all 12 cycles. It was found that 27% of the solidified products did not survive all 12 cycles of the test; the ranges of the number of cycles to failure for solidified products of each waste type are reported in Table 21a. Overall, the number of cycles to failure ranged from one to more than 12,* with a median of 7.

For the solidified products that survived the 12 wet/dry cycles, the ranges of the cumulative corrected relative weight loss after 12 cycles for the solidified products of each raw waste are shown in Table 21b; the median cumulative corrected relative weight loss was 0.95% (w/dw), with a range from -8.6 to 28%.

The overall-pooled interlaboratory variances were used to calculate 95% confidence intervals for the median number of cycles to failure (7 ± 4 cycles) and the median cumulative corrected relative weight loss after 12 cycles (0.95 ± 6%, w/dw). For the number of cycles to failure and the weight loss at 12 cycles, the observed range exceeded the interlaboratory 95% confidence interval on the median, showing that real differences between solidified products were measured. In this respect, the wet/dry test differed from the freeze/thaw test, which did not show a definite difference between the weight loss at 12 cycles for different solidified products.

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* See footnote on p. 33.

Table 21 Summary of Wet/Dry Test Results

(a) Solidified products that did not survive 12 cycles

Raw Waste	Number of Products Tested	Number Not Surviving 12 Cycles	Number of Cycles to Failure Median	Range
WTC synthetic solution	12	2	1	1
WES synthetic sludge	14	6	7	1 to 11
Aluminum coil plating sludge	13	4	10.1	3.3 to 12.5
Wood preservation waste soil	13	2	7.7	3 to 12.3
Overall	52	14	7	1 to 12.5

Summary of Statistics	Standard Deviation	95% C.I. for Median
Intralaboratory	2.6	-
Interlaboratory	2.1	3 to 11

Note: C.I., confidence interval.

(b) Solidified products that survived 12 cycles

Raw Waste	Number Surviving 12 Cycles	Weight Loss After 12 Cycles (%, w/dw) Median	Range
WTC synthetic solution	10	0.83	-8.6 to 28
WES synthetic sludge	8	1.67	0.68 to 6.5
Aluminum coil plating sludge	9	0.16	-3.9 to 2.1
Wood preservation waste soil	11	0.33	0.83 to 1.7
Overall	38	0.95	-8.6 to 28

Summary of Statistics	Standard Deviation	95% C.I. for Median
Intralaboratory	2.2	-
Interlaboratory	3.0	-5.0 to 6.9

Note: A negative weight loss indicates greater control specimen weight loss than test specimen weight loss.
C.I., confidence interval.

The overall intralaboratory and interlaboratory variances were compared using an F test at the 1% significance level. For both the number of cycles to failure and the weight loss after 12 cycles, the variances within and between laboratories were not significantly different.

The laboratory pair data comparison plots for the number of cycles to failure and the weight loss after 12 cycles show scattering. For the number of cycles to failure, the correlation coefficient between Wastewater Technology Centre and Alberta Environmental Centre data is 0.9, but the correlation between Alberta Environmental Centre and Waterways Experiment Station data is much weaker, with a coefficient of 0.3. Wastewater Technology Centre and Waterways Experiment Station data could not be correlated, since these two laboratories had too few points in common. For the weight loss after 12 cycles, correlation coefficients for the three pairs of laboratories ranged from 0.3 to 0.5. Again, a t-test at the 1% probability level showed no significant bias in any of the laboratories for the number of cycles to failure or for the cumulative weight loss after 12 cycles.

5.2 Chemical Evaluation Results

As was done for the physical tests, the tables in the following sections summarize the interlaboratory average values of the parameters as medians and ranges, across the solidified products. For each parameter, the overall-pooled intralaboratory standard deviation, the overall-pooled interlaboratory standard deviation, and the 95% confidence interval on the median of the range (based on the interlaboratory standard deviation) are also presented. The recommended number of replicates for each parameter in each of the tests is summarized in Section 7.

5.2.1 Equilibrium Extraction

For analytical data, the standard deviation is commonly a function of the magnitude of the concentrations; therefore, the statistical analysis was performed on the logarithmic transformations of the data. Geometric average concentrations were calculated for those solidified products for which the concentrations were above detection for all replicates in all laboratories. Table 22 summarizes these results for the contaminants in the solidified products of each raw waste type.

An F test at the 1% significance level was used to compare the overall-pooled interlaboratory variance with the overall-pooled intralaboratory variance for the concentrations of each contaminant.

The 95% confidence intervals for the median value of each parameter, calculated using the overall-pooled interlaboratory variances, are summarized in Table 23, along with the intralaboratory and interlaboratory standard deviations.

The variability of the contaminant concentrations in the equilibrium extracts for the solidified products of each of the raw wastes is discussed in the following sections.

Wastewater Technology Centre synthetic solution. No interlaboratory analysis could be conducted for the results from the Wastewater Technology Centre synthetic solution solidified products, as the equilibrium extraction was applied to each solidified product in only one laboratory. Therefore, no comparison could be made between the interlaboratory variance and the intralaboratory variance, or the observed range of values.

Waterways Experiment Station synthetic sludge. For the equilibrium extractions of the solidified Waterways Experiment Station synthetic sludge, the interlaboratory variance was significantly greater than the intralaboratory variance for pH, cadmium, chromium, and nickel. For mercury, there was no significant difference between the interlaboratory and intralaboratory variances.

Comparing the range of contaminant concentrations observed in the extracts with the interlaboratory 95% confidence interval showed that no real difference between solidified products was observed for pH and nickel. For chromium, some measured concentrations exceeded the high end of the 95% confidence interval, and for mercury, some measured concentrations were outside the low end of the 95% confidence interval on

Table 22 **Summary of Concentrations Measured in the Equilibrium Extraction for All Solidified Products**

Raw Waste	Parameter	Number of Products Tested	Number Above Detection*	Concentration in Extract (mg/L)			
				Median	Range		
WTC synthetic solution	pH	14	14	12.0	10.3	to	12.7
	Arsenic	14	14	0.25	0.010	to	1.2
	Cadmium	14	3	0.010	0.007	to	0.011
	Chromium	14	13	0.11	0.022	to	5.8
	Lead	14	10	0.47	0.003	to	11.4
	Phenol	14	14	310	2.1	to	1880
WES synthetic sludge	pH	14	14	12.1	11.7	to	12.8
	Cadmium	14	1	0.0014	-		
	Chromium	14	14	0.083	0.012	to	2.4
	Nickel	14	10	0.022	0.008	to	0.099
	Mercury	14	14	1.2	0.001	to	5.4
Aluminum coil plating sludge	pH	13	13	11.2	8.6	to	12.5
	Arsenic	13	3	0.0018	0.0010	to	0.0028
	Chromium	13	13	0.28	0.021	to	1.18
	Lead	13	3	0.018	0.015	to	0.033
	Thallium	13	0	-	-		
	Total cyanide	13	13	11.9	0.027	to	325
Dredged sediment	pH	15	15	11.9	7.0	to	12.9
	Chromium	15	14	0.048	0.011	to	0.35
	Copper	15	12	1.1	0.14	to	4.0
	Lead	15	6	0.034	0.021	to	0.089
	Mercury	15	5	0.00034	0.00021	to	0.00090
	Zinc	15	9	0.059	0.028	to	0.45
	Polychlorinated biphenyls	15	0	-	-		
	Total organic carbon	15	15	251	93	to	365
Wood preservation waste soil	pH	13	13	11.7	4.6	to	12.5
	Arsenic	13	5	0.0013	0.00089	to	0.0023
	Lead	13	1	0.0068	-		
	Mercury	13	5	0.00018	0.00012	to	0.0019
	Pentachlorophenol	13	13	97	5.3	to	317
	Polyaromatic hydrocarbons	13	0	-	-		
	Total organic carbon	13	13	442	94	to	824

Note: -, data not available.

* "Number above detection" refers to the number of solidified products in which the concentration of the parameter was above detection for all replicates in all laboratories.

the median, indicating that for these contaminants, some measured concentrations were, respectively, greater and less than would be expected based solely on experimental error. Cadmium was below detection in the majority of the leachates.

Aluminum coil plating sludge. The interlaboratory variance was significantly greater than the intralaboratory variance for pH, and chromium, lead, and total cyanide concentrations in the extracts from the aluminum coil plating sludge solidified products.

Comparison of the measured range of contaminant concentrations in the extracts with the range predicted by the interlaboratory 95% confidence interval showed that for pH, chromium, and cyanide some measured concentrations were outside the low end of the 95% confidence interval. The majority of arsenic, lead, and thallium concentrations were below detection in the extracts, probably because the concentrations of these contaminants were quite low in the raw waste initially.

Dredged sediment. For the solidified sediment, the interlaboratory variances for pH, and chromium, copper, lead, and zinc concentrations in the equilibrium extracts were greater than the intralaboratory variances. No significant difference was noted between the interlaboratory and intralaboratory variances for mercury or total organic carbon.

Table 23 **Summary of Statistics for Concentrations Measured in the Equilibrium Extraction***

Raw Waste	Parameter	Standard Deviation Intralaboratory	Interlaboratory	95% C.I. for Median
WTC synthetic solution	pH	0.49	-	-
	Arsenic	35%	-	-
	Cadmium	19%	-	-
	Chromium	26%	-	-
	Lead	517%	-	-
	Phenol	6%	-	-
WES synthetic sludge	pH	0.06	0.51	11.1 to 13.1
	Cadmium	145%	713%	**
	Chromium	41%	195%	0.0086 to 0.80
	Nickel	51%	280%	0.0011 to 0.42
	Mercury	255%	188%	0.13 to 11.0
Aluminum coil plating sludge	pH	0.48	0.75	9.7 to 12.7
	Arsenic	151%	272%	**
	Chromium	35%	188%	0.030 to 2.57
	Lead	74%	751%	**
	Thallium	-	-	-
	Total cyanide	35%	713%	0.15 to 974
Dredged sediment	pH	0.29	1.0	9.9 to 13.9
	Chromium	38%	223%	0.0041 to 0.56
	Copper	78%	272%	0.071 to 16.9
	Lead	58%	379%	0.00094 to 1.2
	Mercury	317%	307%	0.00002 to 0.0074
	Zinc	55%	163%	0.0069 to 0.51
	Polychlorinated biphenyls	-	-	-
	Total organic carbon	41%	51%	97 to 647
Wood preservation waste soil	pH	0.29	0.67	10.4 to 13.0
	Arsenic	26%	26%	**
	Lead	91%	-	-
	Mercury	66%	1413%	**
	Pentachlorophenol	51%	78%	27 to 346
	Polyaromatic hydrocarbons	29%	-	-
	Total organic carbon	7%	23%	283 to 691

Note: -, data not available.
C.I., confidence interval.

* For a pair of measurements in each laboratory.

** Meaningful confidence intervals could not be calculated if there were fewer than eight degrees of freedom.

The observed range of concentrations in the extracts was smaller than the interlaboratory 95% confidence interval for chromium, copper, lead, mercury, and zinc, showing that for these contaminants no difference between solidified products was measured. For pH and total organic carbon, some measured concentrations were lower than the lower limit of the 95% confidence interval. The concentration of polychlorinated biphenyls was below the analytical detection limit for all the extracts, probably because the concentration of polychlorinated biphenyls was very low in the raw waste.

Wood preservation waste soil. The interlaboratory variance was significantly greater than the intralaboratory variance for pH, and pentachlorophenol and total organic carbon concentrations in the extracts for the wood preservation waste soil solidified products.

Comparing the range of observed contaminant concentrations with the interlaboratory 95% confidence interval showed that the observed range was wider than the confidence interval for total organic carbon, and that some pH values and pentachlorophenol concentrations were outside the low end of the 95% confidence interval on the median. Concentrations of arsenic, lead, mercury, and polyaromatic hydrocarbons were largely below detection, again probably because the concentrations of these contaminants were quite low in the raw waste.

Summary. In general, the ranges of concentrations measured in the equilibrium extraction were close to the ranges defined by the 95% confidence intervals. The inability of the equilibrium extraction to show clear differences between solidified products is a consequence of two factors: 1) the similarity of immobilization mechanisms in the different solidified products, and 2) poor analytical reproducibility in this low concentration range.

Although the laboratory pair plots for the concentrations measured in the equilibrium extraction show data above and below detection for all the wastes on one plot for each contaminant, the correlation coefficients were calculated separately for the solidified products of each waste, using measurements above detection only. Overall, the correlation coefficients ranged from 0.05 to 0.98; many plots show extensive scattering.

The paired t-test comparison of the contaminant concentrations in the laboratory pairs showed a statistically significant difference between chromium concentrations measured by the Wastewater Technology Centre and the Alberta Environmental Centre in the extracts from the Waterways Experiment Station synthetic sludge samples at the 1% significance level; however, there did not appear to be a bias in the measurements for any of the other contaminants in any of the three laboratories.

5.2.2 U.S. EPA Toxicity Characteristic Leaching Procedure (TCLP)

As was the case for the data from the equilibrium extraction, the statistical analysis was performed on the logarithmic transformations of the data. Geometric average concentrations were calculated for those solidified products for which the concentrations were above detection for all replicates in all laboratories. Table 24 summarizes these results for the contaminants in the solidified products of each raw waste type.

An F test was used to compare the overall-pooled interlaboratory variance with the overall-pooled intralaboratory variance for the concentrations of each contaminant.

The overall-pooled interlaboratory variance was used to calculate the 95% confidence intervals for the median value of each parameter presented in Table 25, along with the intralaboratory and interlaboratory standard deviations.

The variability of the contaminant concentrations in the toxicity characteristic leaching procedure leachates is discussed for the solidified products of each of the raw wastes in the following sections.

Wastewater Technology Centre synthetic solution. No interlaboratory analysis could be conducted for the results from the Wastewater Technology Centre synthetic solution solidified products, as the toxicity characteristic leaching procedure was applied to each solidified product in only one laboratory. Therefore, no comparison could be made between the interlaboratory variance and the intralaboratory variance, or the observed range of values.

Waterways Experiment Station synthetic sludge. For the toxicity characteristic leaching procedure results for the solidified Waterways Experiment Station synthetic sludge, the interlaboratory variance was significantly greater than the intralaboratory variance for pH, chromium, and mercury.

Comparing the range of contaminant concentrations observed in the extracts with the interlaboratory 95% confidence interval showed that the observed range exceeded the confidence interval for pH. For chromium, some measured concentrations exceeded the high end of the 95% confidence interval, and for mercury, some measured concentrations were outside the low end of the 95% confidence interval on the median, indicating that for these contaminants, some measured concentrations were, respectively, greater and

Table 24 Summary of Concentrations Measured in the Toxicity Characteristic Leaching Procedure for All Solidified Products

Raw Waste	Parameter	Number of Products Tested	Number Above Detection*	Concentration in Extract (mg/L)			
				Median	Range		
WTC synthetic solution	pH	14	14	9.9	5.6	to	12.7
	Arsenic	14	14	0.17	0.0087	to	36.4
	Cadmium	14	8	0.099	0.003	to	31.5
	Chromium	14	9	0.12	0.030	to	1.34
	Lead	14	7	0.046	0.0032	to	4.3
	Phenol	14	14	43.9	0.073	to	144
WES synthetic sludge	pH	14	14	10.1	7.8	to	12.5
	Cadmium	14	2	-	0.067	to	0.091
	Chromium	14	14	0.052	0.012	to	5.9
	Nickel	14	1	0.34	-		
	Mercury	14	11	0.51	0.001	to	3.1
Aluminum coil plating sludge	pH	13	13	8.7	5.2	to	10.7
	Arsenic	13	1	0.0017	-		
	Chromium	13	2	-	1.4	to	4.5
	Lead	13	0	-	-		
	Thallium	13	0	-	-		
	Total cyanide	13	13	7.0	1.8	to	29
Dredged sediment	pH	15	15	7.8	4.5	to	11.6
	Chromium	15	4	0.020	0.0078	to	0.30
	Copper	15	12	0.31	0.079	to	0.79
	Lead	15	0	-	-		
	Mercury	15	5	0.0065	0.00028	to	0.013
	Zinc	15	12	0.16	0.085	to	19.7
	Polychlorinated biphenyls	15	0	-	-		
Wood preservation waste soil	pH	13	13	8.1	4.7	to	10.2
	Arsenic	13	2	-	0.0032	to	0.031
	Lead	13	1	0.011	-		
	Mercury	13	0	-	-		
	Pentachlorophenol	13	13	28	3.0	to	162
	Polyaromatic hydrocarbons	13	0	-	-		

Note: -, data not available.

* "Number above detection" refers to the number of solidified products in which the concentration of the parameter was above detection for all replicates in all laboratories.

less than would be expected based solely on experimental error. Cadmium and nickel were below detection in the majority of the leachates.

Aluminum coil plating sludge. The interlaboratory variance was significantly greater than the intralaboratory variance for pH, and arsenic, chromium, and total cyanide concentrations in the toxicity characteristic leaching procedure extracts from the aluminum coil plating sludge solidified products.

Comparison of the measured range of contaminant concentrations in the extracts with the range predicted by the interlaboratory 95% confidence interval showed that the observed range of cyanide concentrations exceeded the limits of the confidence interval. Some pH values were outside the low end of the 95% confidence interval. The majority of arsenic, chromium, lead, and thallium concentrations were below detection. In the cases of arsenic, lead, and thallium, the explanation for this observation may be the very low concentration of these contaminants in the solidified waste initially.

Dredged sediment. For the solidified sediment, the interlaboratory variances for pH, and chromium, copper, mercury, and zinc concentrations in the toxicity characteristic leaching procedure extracts were greater than the intralaboratory variances.

The observed range of concentrations in the extracts was wider than the interlaboratory 95% confidence interval for pH, showing that a difference between solidified products was measured. For mercury some measured concentrations were lower than the lower

Table 25 **Summary of Statistics for Concentrations Measured in the Toxicity Characteristic Leaching Procedure**

Raw Waste	Parameter	Standard Deviation		95% C.I. for Median		
		Intralaboratory	Interlaboratory			
WTC synthetic solution	pH	0.46	-	-		
	Arsenic	298%	-	-		
	Cadmium	81%	-	-		
	Chromium	13%	-	-		
	Lead	57%	-	-		
	Phenol	13%	-	-		
WES synthetic sludge	pH	0.02	1.0	7.9	to	12.3
	Cadmium	122%	64%	•		
	Chromium	42%	153%	0.0067	to	0.40
	Nickel	84%	132%	•		
	Mercury	41%	224%	0.034	to	7.6
Aluminum coil plating sludge	pH	0.09	0.77	5.8	to	13.1
	Arsenic	34%	568%	•		
	Chromium	52%	409%	•		
	Lead	12%	-	-		
	Thallium	-	-	-		
	Total cyanide	18%	43%	3.2	to	15.4
Dredged sediment	pH	0.2	1.2	6.3	to	11.1
	Chromium	137%	321%	•		
	Copper	43%	95%	0.076	to	1.3
	Lead	1044%	-	-		
	Mercury	144%	1119%	0.00034	to	0.12
	Zinc	76%	306%	0.0085	to	3.0
	Polychlorinated biphenyls	-	-	-		
Wood preservation waste soil	pH	0.29	0.95	6.2	to	10.0
	Arsenic	10%	114%	•		
	Lead	11%	558%	•		
	Mercury	98%	-	-		
	Pentachlorophenol	72%	78%	7.8	to	100.8
	Polyaromatic hydrocarbons	-	-	-		

Note: -, data not available.
C.I., confidence interval.
• Meaningful confidence intervals could not be calculated if there were fewer than eight degrees of freedom.

limit of the 95% confidence interval; whereas for zinc, some measured concentrations exceeded the upper limit of the 95% confidence interval. The concentrations of chromium, lead, and polychlorinated biphenyls were below the analytical detection limit for all the extracts.

Wood preservation waste soil. The interlaboratory variance was significantly greater than the intralaboratory variance for pH, and arsenic and lead concentrations in the toxicity characteristic leaching procedure extracts for the wood preservation waste soil solidified products. No significant difference was noted between the interlaboratory and intralaboratory variances for pentachlorophenol concentrations.

Comparing the range of observed contaminant concentrations with the interlaboratory 95% confidence interval showed that the observed range was wider than the confidence interval for pH and pentachlorophenol. Concentrations of arsenic, lead, mercury, and polyaromatic hydrocarbons were largely below detection.

Summary. In general, for the toxicity characteristic leaching procedure, observed parameter ranges were slightly wider than were seen in the equilibrium extraction because of the wider range of leachate pHs caused by the differing acid neutralization capacities of the solidified products. The differences between solidified products were often still insufficient to prevail over the relatively poor analytical reproducibility that is typical of the low concentration range. In a number of instances, metals that were detectable in the equilibrium extracts were undetected in the toxicity characteristic leaching procedure extracts. Since metals are amphoteric, the acid addition of the toxicity characteristic leaching procedure may actually have decreased the solubility of the metal compounds by lowering high extract pHs only slightly.

The laboratory pair plots for the concentrations measured in the toxicity characteristic leaching procedure again show measurements below detection; however, these measurements have not been included in calculating the correlation coefficients. The correlation coefficients ranged from 0.16 to 0.99.

A paired t-test comparison of the contaminant levels measured by the laboratory pairs showed that at the 1% significance level there was a statistically significant difference in cyanide levels measured at the Alberta Environmental Centre and the Waterways Experiment Station in the leachates from the aluminum coil plating sludge solidified products. This was the only apparent bias detected for all the contaminants.

5.2.3 *Acid Neutralization Capacity*

The results of determining the acid neutralization capacity of each of the solidified products for the Wastewater Technology Centre synthetic solution and the Waterways Experiment Station synthetic sludge are summarized in Table 26, along with the results of the statistical analysis. The test method required choosing a schedule of 11 acid additions, based on a pretest, from three possible acid addition schedules, with final acid additions of 4, 8, and 12 meq of nitric acid per gram of dry waste respectively. Schedule 1 was applied to two solidified products, schedule 2 was applied to 10 solidified products, and schedule 3 was applied to 21 solidified products. For five of the 14 Waterways Experiment Station synthetic sludge solidified products, the pretest resulted in choosing different acid addition schedules in the two laboratories. The statistical analysis was conducted only on the results from solidified products for which the acid addition schedule was the same. Table 26 shows the median pH and the pH ranges for four of the acid additions: 1) 0 meq/g, which was common to all three schedules; 2) 2.4 meq/g, which was also common to all three schedules; 3) 7.2 meq/g, which was added in schedules 2 and 3 only; and 4) 12 meq/g, which was added in schedule 3 only. For an acid addition of 0 meq/g, the pHs measured ranged from 10.7 to 13.2, with a median of 12.2; for 2.4 meq/g, pHs ranged from 5.5 to 12.7, with a median of 10.9; for 7.2 meq/g, pHs ranged from 0.8 to 11.3, with a median of 7.2; and for 12 meq/g, the pHs ranged from 1.1 to 8.8, with a median of 4.2.

An F test was used to compare the overall-pooled interlaboratory variance with the overall-pooled intralaboratory variance.

Table 26 **Summary of Acid Neutralization Capacity Determination Results**

	Number of Products Tested				Amount of Nitric Acid Added (meq/g of dry waste)							
	Schedule				0		2.4		7.2		12	
					pH		pH		pH		pH	
Raw Waste	1	2	3	Total*	Median	Range	Median	Range	Median	Range	Median	Range
WTC synthetic solution	2	4	8	14	12.3	10.7 to 12.9	10.8	5.5 to 12.1	7.2	2.6 to 10.8	5.1	1.1 to 8.8
WES synthetic sludge	0	6	13	141	12.2	11.7 to 13.2	11.0	6.6 to 12.7	7.2	0.8 to 11.3	3.3	2.5 to 8.1
Overall	2	10	21	28	12.2	10.7 to 13.2	10.9	5.5 to 12.7	7.2	0.8 to 11.3	4.2	1.1 to 8.8

Standard Deviation					
Intralaboratory**	Interlaboratory***	95% C.I. for Median	95% C.I. for Median	95% C.I. for Median	95% C.I. for Median
0.77	1.18	9.8 to 14.6	8.5 to 13.3	4.8 to 9.6	1.8 to 6.6

Note: C.I., confidence interval.

* The total is not necessarily the sum of the number of products tested using schedules 1, 2, and 3, since some solidified products were tested using different acid addition schedules in the different laboratories.

** For a single pH measurement.

*** For a group of two pH measurements in each laboratory.

The interlaboratory variance was greater than the intralaboratory variance at the 1% significance level.

The 95% confidence intervals for the median pHs for each of the four acid additions, calculated using the overall-pooled interlaboratory variance, were 12.2, 10.9, 7.2, and 4.2 ± 2.4. The observed range of pHs for the different solidified products may be compared with the interlaboratory 95% confidence interval for each of the four acid additions. It can be seen that for 0 meq/g, the range of pHs described by the 95% confidence interval on the median pH exceeds the observed range for the different solidified products. For 7.2 and 12 meq/g, it can be seen that the observed range exceeds the 95% confidence interval. Thus, a difference between solidified products was measured only for higher acid additions. This is logical if we consider that cement-based solidified products have similar pHs if there is no acid addition, but may have different pHs after acid addition, depending upon their different acid neutralization capacities (i.e., amount of excess lime).

The laboratory pair plots for the average pHs measured for each solidified product in each laboratory show that the data points approximate a line with the equation $x = y$. The correlation coefficients for these data points are 0.8 or greater, indicating little scattering from a straight line. A t-test comparison of the average difference between the laboratory pairs with a hypothetical difference of zero shows that there is no significant difference between average pHs measured at the Wastewater Technology Centre and the Waterways Experiment Station, or between pHs measured at the Alberta Environmental Centre and the Waterways Experiment Station at the 1% level. However, pHs measured at the Alberta Environmental Centre are significantly higher than those measured at the Wastewater Technology Centre by an average of 0.8.

5.2.4 Sequential Chemical Extraction

The percentage of each contaminant extracted in each of the five fractions of the sequential chemical extraction was calculated by dividing the amount of contaminant extracted in each fraction (in milligrams per gram of dry waste) by the total calculated contaminant concentration in the solidified product. This calculated total contaminant concentration was used, rather than the measured or additive total, to provide a common basis for comparing the results from the different laboratories, and to make the percentage in each fraction independent of the amounts extracted in the other fractions. The total percentage extracted, therefore, may be greater or less than 100%.

Once again, the statistical analysis was performed on the logarithmic transformations of the data. Geometric average percentages were calculated for those fractions for which the concentrations were above detection for all replicates. Table 27 summarizes these results for the contaminants in the solidified products of each raw waste type. The median and range for each fraction are shown, as well as the median and range for the total percentage extracted.

Subtracting 100% from the total percentage extracted results in the percentage difference between the calculated total concentration in the sample and the total concentration measured in the sequential chemical extraction. Based on the median total percentages extracted, it appears that the sequential chemical extraction most often overestimated the amount of contaminant in the sample by 2 to 86% with respect to the calculated contaminant concentration, with the exception of arsenic in the aluminum coil plating sludge solidified products, which tended to be underestimated.

An F test was used to compare the overall-pooled interlaboratory variance with the overall-pooled intralaboratory variance for

Table 27 Summary of Contaminant Percentages Extracted in the Fractions of the Sequential Chemical Extraction

Raw Waste	Parameter	Fraction	Number of Products Tested	Number Above Detection*	Percentage in Fraction			
					Median	Range		
WTC synthetic solution	Arsenic	A	14	14	0.25	0.11	to	2.0
		B	14	14	41	1.5	to	87
		C	14	14	50	5.1	to	198
		D	14	14	2.7	0.14	to	24
		E	10	10	1.9	0.78	to	11
		Total	10		134	102	to	177
	Cadmium	A	14	7	0.19	0.062	to	0.89
		B	14	14	86	1.4	to	123
		C	14	14	11	3.4	to	133
		D	14	13	1.1	0.19	to	36
		E	14	13	0.12	0.056	to	1.6
		Total	14		108	62	to	142
	Chromium	A	14	0	-	-		
		B	14	14	65	1.6	to	130
		C	14	13	32	7.3	to	191
		D	14	14	5.9	0.23	to	31
		E	14	14	7.5	1.7	to	345
		Total	14		156	103	to	435
	Lead	A	14	0	-	-		
		B	14	12	57	20	to	88
		C	14	14	41	7.7	to	105
		D	14	12	2.5	0.15	to	27
		E	14	14	1.1	0.47	to	24
		Total	14		102	57	to	135
WES synthetic sludge	Cadmium	A	14	3	0.11	0.06	to	0.32
		B	14	14	5.6	0.01	to	145
		C	14	14	82	7.0	to	226
		D	14	14	1.7	0.12	to	95
		E	14	12	0.13	0.01	to	0.63
		Total	14		132	91	to	229
	Chromium	A	14	11	0.099	0.055	to	0.71
		B	14	14	9.9	0.079	to	136
		C	14	14	107	26	to	220
		D	14	14	2.7	0.26	to	16
		E	14	14	0.49	0.02	to	3.7
		Total	14		138	89	to	223
	Nickel	A	14	7	0.089	0.01	to	0.16
		B	14	11	2.2	0.01	to	96
		C	14	14	123	39	to	243
		D	14	14	2.4	0.17	to	10
		E	14	14	0.28	0.014	to	2.5
		Total	14		129	82	to	246
	Mercury	A	14	13	0.57	0.05	to	12
		B	14	13	6.0	0.048	to	102
		C	7	6	17.8	0.91	to	34
		D	14	13	26	11	to	380
		E	14	13	3.3	0.47	to	42
		Total	7		118	44	to	218
Aluminum coil plating sludge	Arsenic	A	11	3	0.82	0.76	to	5.9
		B	11	11	3.5	1.0	to	132
		C	11	11	28	0.96	to	485
		D	10	6	17	2.3	to	39
		E	3	3	20	1.2	to	45
		Total	3		58	41	to	92
	Chromium	A	12	3	0.51	0.37	to	2.0
		B	12	12	41	1.4	to	67
		C	12	12	33	6.1	to	87
		D	12	10	6.6	1.4	to	38
		E	12	11	8.2	0.76	to	155
		Total	12		105	71	to	230
	Lead	A	12	0	-	-		
		B	12	6	47	0.83	to	165
		C	12	4	19	3.4	to	303
		D	12	3	2.0	1.7	to	4.0
		E	12	9	36	6.4	to	452
		Total	12		186	10	to	416
	Thallium	A	12	5	-	-		
		B	12	0	-	-		
		C	12	0	-	-		
		D	12	0	-	-		
		E	12	0	-	-		
		Total	12		0	0		
Dredged sediment	Chromium	A	15	2	-	0.8	to	1.2
		B	15	12	31	1.8	to	106
		C	15	11	55	20	to	187
		D	15	13	17	4.9	to	104
		E	13	13	44	6.7	to	453
		Total	13		151	82	to	469
	Copper	A	15	4	8.1	6.1	to	11
		B	15	12	41	6.7	to	157
		C	15	14	41	9.5	to	131
		D	15	13	45	29	to	183
		E	14	13	41	8.3	to	83
		Total	14		171	56	to	455
	Lead	A	15	0	-	-		
		B	15	7	79	31	to	153
		C	15	13	77	9.9	to	130
		D	15	8	11	3.5	to	19
		E	14	13	37	9.7	to	140
		Total	14		148	79	to	250
	Zinc	A	15	2	-	0.51	to	0.74
		B	15	15	55	4.3	to	107
		C	15	14	23	4.5	to	87
		D	15	15	7.1	3.6	to	12
		E	14	14	19	13	to	38
		Total	14		113	85	to	164

Note: -, data not available.

the concentrations of each contaminant. As no interlaboratory variance could be calculated for the results from the Waterways Experiment Station synthetic sludge solidified products, no F-test comparison could be made for these. The interlaboratory variance was significantly greater than the intralaboratory variance at the 1% level for most contaminants in most fractions of the test.

The interlaboratory variance, calculated for the Wastewater Technology Centre and the Alberta Environmental Centre only, since this test was not performed by the Waterways Experiment Station, was used to calculate a 95% confidence interval for the median value of each contaminant percentage, as summarized in Table 28, along with the intralaboratory and interlaboratory standard deviations. Confidence intervals were calculated for the contaminants present in the Wastewater Technology Centre synthetic solution solidified products only, since the Waterways Experiment Station synthetic sludge solidified products were each tested in only one laboratory, and there were too few degrees of freedom to allow for the calculation of meaningful confidence intervals for the aluminum coil plating sludge and dredged sediment contaminants. For the Wastewater Technology Centre synthetic solution solidified products, comparing the range of contaminant percentages in each fraction with the 95% interlaboratory confidence intervals for these percentages showed that in most cases the confidence interval was wider than the observed range. Therefore, no statistically significant difference between the Wastewater Technology Centre synthetic solution solidified products was observed.

No laboratory pair plots were constructed for these data. The correlation coefficients ranged from 0.03 to 0.98.

Comparison of the measurements in both laboratories using a paired t-test showed an average difference that was statistically significant at the 1% level for arsenic in fraction B, cadmium in fraction E, chromium in fractions B and E, and lead in fractions B and E for the Wastewater Technology Centre synthetic solution solidified products; chromium in fraction D and lead in fraction E for the aluminum coil plating sludge solidified products; and zinc in fraction D for the dredged sediment solidified products.

5.2.5 Dynamic Leach Test

The dynamic leach test was applied to the solidified products of the Wastewater Technology Centre synthetic solution, the aluminum coil plating sludge, and the wood preservation waste soil.

For each contaminant in each waste, the ranges of the average leachability indices determined using the dynamic leach test are summarized in Table 29. If the data from only one of the laboratories were below detection (which may have occurred if the detection limit in that laboratory was higher than in the other), the data below detection were not considered. In the following sections, the statistical analysis of the measured leachability indices is discussed. In addition, the results from the dynamic leach test are analyzed to determine the validity of the assumptions inherent to the model upon which the test is based.

Analysis of reproducibility. The overall-pooled intralaboratory variance was calculated using the individual leachability indices from each leaching interval, since replicates of the entire test (involving seven or nine leaching intervals each, depending on the schedule chosen) were not conducted within the two laboratories. The overall-pooled interlaboratory variance was calculated using averaged data from the Wastewater Technology Centre and the Alberta Environmental Centre only because the dynamic leach test was not conducted by the Waterways Experiment Station.

An F test, at the 1% significance level, was used to compare the interlaboratory variance with the overall-pooled intralaboratory

Table 28 Summary of Statistics for Contaminant Percentages Extracted in the Fractions of the Sequential Chemical Extraction

Raw Waste	Parameter	Fraction	Percent Standard Deviation		95% C.I. for Median
			Intralaboratory	Interlaboratory	
WTC synthetic solution	Arsenic	A	202	96	0.057 to 1.1
		B	44	223	3.1 to 541
		C	72	158	6.2 to 405
		D	92	298	0.13 to 57
		E	56	-	-
	Cadmium	A	77	283	*
		B	283	345	3.2 to 2322
		C	30	85	2.8 to 43
		D	74	180	0.11 to 11
		E	86	393	0.004 to 4.0
	Chromium	A	22	-	-
		B	33	173	7.1 to 591
		C	63	115	5.9 to 173
		D	52	187	0.58 to 60
		E	30	*469	0.16 to 345
	Lead	A	55	-	-
		B	33	129	9.2 to 353
		C	33	57	15 to 111
		D	158	216	0.20 to 32
		E	79	337	0.043 to 28
WES synthetic sludge	Cadmium	A	11	-	-
		B	434	-	-
		C	40	-	-
		D	33	-	-
		E	58	-	-
	Chromium	A	60	-	-
		B	83	-	-
		C	36	-	-
		D	81	-	-
		E	100	-	-
	Nickel	A	6	-	-
		B	65	-	-
		C	79	-	-
		D	44	-	-
		E	68	-	-
	Mercury	A	166	-	-
		B	329	-	-
		C	60	-	-
		D	59	-	-
		E	55	-	-
Aluminum coil plating sludge	Arsenic	A	38	-	-
		B	33	615	*
		C	47	59	*
		D	52	-	-
		E	144	-	-
	Chromium	A	83	-	-
		B	12	224	*
		C	9	106	*
		D	49	129	*
		E	107	107	*
	Lead	A	-	-	-
		B	151	1280	*
		C	13	-	-
		D	55	-	-
		E	72	832	*
	Thallium	A	-	-	-
		B	-	-	-
		C	-	-	-
		D	-	-	-
		E	-	-	-
Dredged sediment	Chromium	A	20	-	-
		B	24	144	*
		C	48	115	*
		D	68	89	*
		E	90	209	*
	Copper	A	82	-	-
		B	43	107	*
		C	56	78	*
		D	33	29	*
		E	27	67	*
	Lead	A	-	-	-
		B	78	81	*
		C	19	68	*
		D	61	246	*
		E	18	246	*
	Zinc	A	107	-	-
		B	106	180	*
		C	17	151	*
		D	21	14	*
		E	24	30	*

Note: -, data not available.
C.I., confidence interval.

* Meaningful confidence intervals could not be calculated if there were fewer than eight degrees of freedom.

Table 29 **Summary of Leachability Indices for All Solidified Products**

Raw Waste	Contaminant	Number of Products Tested	Number of LXs Calculated*	Median LX	Range of Leachability Indices in the Different Solidified Products
WTC synthetic solution	Arsenic	11	11	11.3	9.2 to 14.7
	Cadmium	11	3	13.8	11.7 to 16.0
	Chromium	11	8	12.2	10.3 to 13.3
	Lead	11	9	12.7	9.1 to 14.5
	Phenol	11	11	7.7	6.4 to 11.8
Aluminum coil plating sludge	Arsenic	11	0	-	-
	Chromium	11	11	11.6	9.6 to 13.0
	Lead	11	9	9.1	7.1 to 10.2
	Thallium	11	0	-	-
	Total cyanide	11	11	8.4	6.3 to 11.6
Wood preservation waste soil	Pentachlorophenol	12	11	8.5	7.7 to 10.7

Note: -, data not available
LX, leachability index

* The leachability index could not be calculated if the contaminant concentrations in the leachates from more than one interval were below detection.

variance for the leachability indices for each contaminant. The 95% confidence intervals calculated for the median leachability indices are reported in Table 30, as well as the intralaboratory and interlaboratory standard deviations. Meaningful confidence intervals could not be calculated for some contaminants, as there were not enough measurements common to both laboratories.

Wastewater Technology Centre synthetic solution. For the synthetic solution solidified products, the interlaboratory variance for the leachability indices was greater than the intralaboratory variance for chromium and phenol. For cadmium and lead, there was no statistically significant difference between the two variances.

Comparison of the range of leachability indices observed with the interlaboratory 95% confidence interval on the median was only possible for arsenic and phenol. For these contaminants, the observed range of leachability indices exceeded the range spanned by the confidence interval, indicating that there were real differences in the leachability indices exhibited by the different solidified products.

Aluminum coil plating sludge. The interlaboratory variance was greater than the intralaboratory variance for the leachability indices measured for cyanide. For chromium, there was no significant difference between the intralaboratory and interlaboratory variances.

Comparing the measured range of leachability indices with the interlaboratory 95% confidence interval showed that a real difference in leachability indices for cyanide was measured for the different solidified products. Leachate concentrations for arsenic and thallium were consistently below detection, as a result of the very low concentrations of these elements in the raw waste.

Wood preservation waste soil. The interlaboratory variance on the leachability index for pentachlorophenol was significantly greater than the intralaboratory variance.

The observed range of leachability indices for pentachlorophenol also exceeded the interlaboratory 95% confidence interval on the median leachability index, showing that real differences in leachability indices for this parameter were measured for the different wood preservation waste soil solidified products.

The laboratory pair plots of the leachability indices for the different contaminants show that in most cases the data points are only slightly scattered from the desired straight line. The corresponding correlation

Table 30 **Summary of Statistics for Leachability Indices***

Raw Waste	Contaminant	Standard Deviation Intralaboratory	Interlaboratory	95% C.I. for Median
WTC synthetic solution	Arsenic	0.72	0.44	10.3 to 12.3
	Cadmium	0.83	2.0	**
	Chromium	0.59	0.84	**
	Lead	0.69	0.22	**
	Phenol	0.39	0.82	5.9 to 9.5
Aluminum coil plating sludge	Arsenic	-	-	-
	Chromium	0.81	0.33	**
	Lead	1.3	-	-
	Thallium	-	-	-
	Total cyanide	0.42	0.78	6.6 to 10.2
Wood preservation waste soil	Pentachlorophenol	0.41	0.49	7.4 to 9.6

Note: -, data not available

* For a group of seven or nine leaching intervals (schedule A or B) in each laboratory.

** Meaningful confidence intervals could not be calculated if there were fewer than seven leachability indices common to both laboratories.

coefficients are 0.8 or greater, with the exception of chromium. The correlation coefficient for the chromium leachability indices is 0.17, but only three observations contribute to this statistic. A paired t-test comparison of the average difference between the leachability indices measured at the Wastewater Technology Centre and the Alberta Environmental Centre with a hypothetical difference of zero shows that the leachability index for pentachlorophenol measured by the Wastewater Technology Centre tends to be higher than that measured at the Alberta Environmental Centre by 0.6 at the 1% significance level. Similar t-test comparisons for the leachability indices for the other contaminants show that there is no statistically significant difference between the leachability indices measured in the two laboratories.

Verification of model assumptions. The dynamic leach test is based on the solution of a diffusion model, with the aid of a number of assumptions (Godbee and Joy, 1974):

1) the mobility of a contaminant is limited by diffusion

2) the specimen behaves as a semi-infinite medium, provided that the cumulative fraction leached does not exceed 20%

3) the concentration of a contaminant at the specimen surface is approximately zero

Data analysis was conducted to verify the validity of these assumptions.

The model solution may be expressed as follows:

$$\left(\frac{\Sigma a_n}{A_o}\right)\left(\frac{V}{S}\right) = 2\left(\frac{D_e'}{\pi}\right)^{1/2} t_n^{1/2} \qquad (10)$$

where a_n = contaminant loss during leaching interval n (mg)

A_o = initial amount of contaminant in the specimen (mg)

V = specimen volume (cm^3)

S = specimen surface area (cm^2)

t_n = time at the end of leaching period n

D_e' = apparent diffusion coefficient (cm^2/s)

It can be seen from Equation 10 that a linear relationship between the cumulative fraction leached and the square root of time is a consequence of diffusion control. Thus, the validity of the assumption of diffusion control was verified by plotting the cumulative fraction leached against the square root of time for each contaminant in each solidified product for which data were available for all the leaching intervals. The plots fall into four general categories of leaching mechanisms:

1) diffusion control
2) initial resistance to leaching followed by diffusion control
3) initial washoff followed by diffusion control
4) linear relationship between cumulative fraction leached and time

An illustration of each of the four different mechanisms is shown in Figures 1 to 3.

Diffusion control is apparent for the release of cadmium from both of the solidified products for which sufficient data were available for plotting. It also appeared to be the mechanism for leaching chromium from all but one solidified product, lead from three solidified products, phenol from five solidified products, cyanide from six solidified products, and pentachlorophenol from eight solidified products.

Diffusion control following an initial period of resistance is the apparent release mechanism for phenol from four solidified products, cyanide from four solidified products, lead from two solidified products, and pentachlorophenol from two solidified products.

An initial period of rapid washoff of contaminants, followed by diffusion, is the apparent leaching mechanism for lead from eight solidified products, chromium from one solidified product, and pentachlorophenol from one solidified product.

The fourth type of plot, a linear relationship between the cumulative fraction leached and time, was observed for arsenic in four of the 11 solidified Wastewater Technology Centre synthetic solution products tested. For the remaining solidified products, the release of arsenic appeared to be diffusion controlled for the first few intervals and then linear with time. It is postulated that this leaching mechanism was observed for arsenic because it was mobilized by the reaction of basic calcium arsenite with the carbonates in CO_2 saturated water. Thus, the arsenic release rate was controlled by the rate of renewal of CO_2 saturated leachate (Côté et al., 1987b).

Examination of the cumulative fraction leached after the last interval shows that the majority of specimens do behave as a semi-infinite medium. The cumulative fraction leached exceeded 20% for phenol from four solidified products, cyanide from one solidified product, and lead from one solidified product. As a consequence, the assumption that the specimen approximated a semi-infinite medium was violated, and the apparent diffusion coefficient was calculated using a dimensionless time factor, specific to the cumulative fraction leached and the length-to-diameter ratio of the cylindrical specimen (ANS, 1986).

Since the difference between a contaminant's concentration in the leachant and its equilibrium concentration provides the driving force for mass transfer, the model's assumption of a zero surface concentration assumes the maximum driving force. This driving force becomes zero when the equilibrium concentration of the contaminant is attained in the leachant. Therefore, it is important to keep the system far from equilibrium. The schedules for leachant renewal were chosen as a compromise between this need and the requirement of a contaminant concentration greater than the detection limit for chemical analysis. Comparison of the average contaminant concentrations in the leachates from the dynamic leach test with the saturation concentrations found in the extracts from the equilibrium extractions for each solidified product shows that equilibrium was not approached during the dynamic leach test leaching intervals for arsenic, chromium,

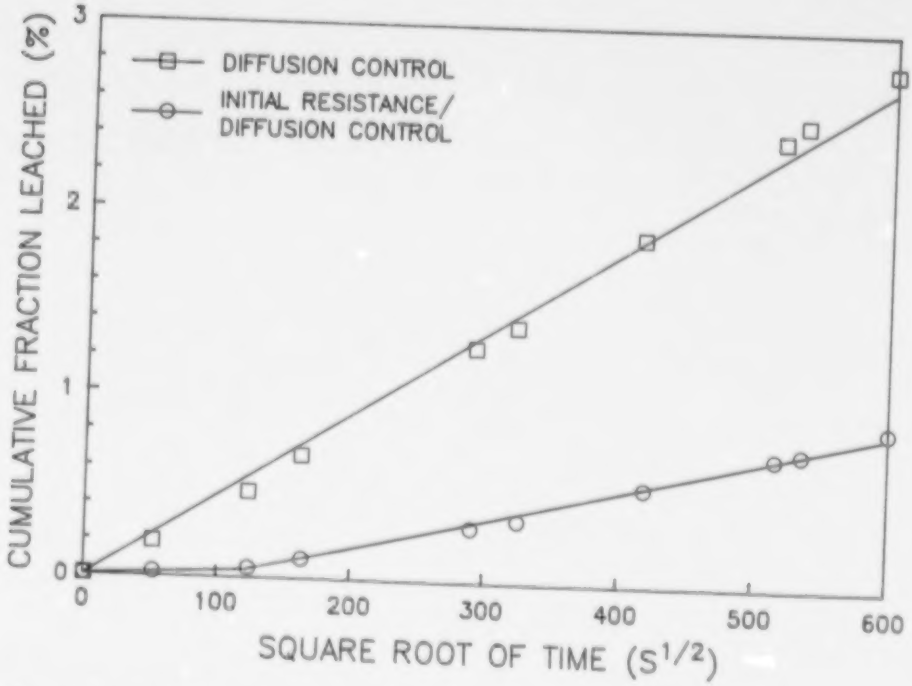

Figure 1 Dynamic Leach Test Data Showing the Diffusion Controlled Release of Cyanide (solidified product IHIB) and Diffusion Control Following Initial Resistance to Leaching (solidified product KAHG)

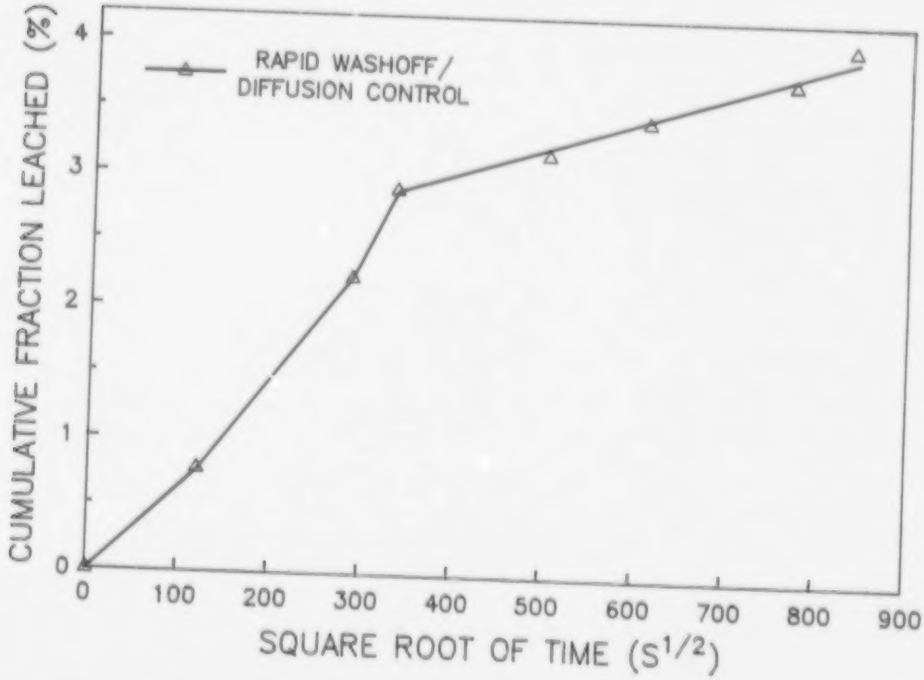

Figure 2 Dynamic Leach Test Data Showing the Diffusion Controlled Release of Lead Following Initial Rapid Washoff (solidified product FDIA)

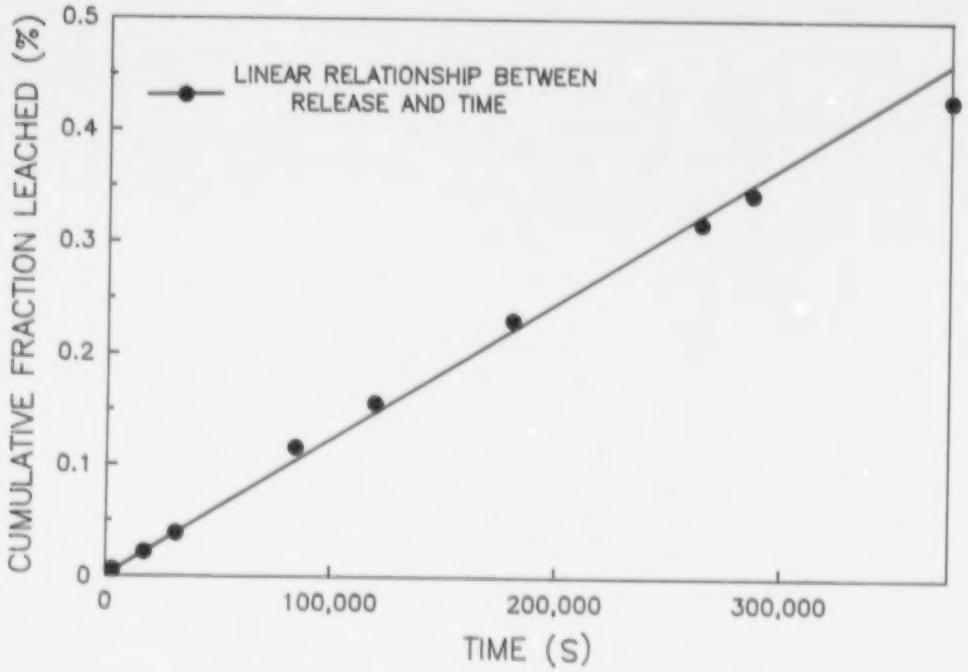

Figure 3 **Dynamic Leach Test Data Showing a Linear Relationship Between the Amount of Arsenic Leached and Time (solidified product EEBH)**

lead, and phenol in the Wastewater Technology Centre synthetic solution solidified products; chromium and cyanide in the aluminum coil plating sludge solidified products; and pentachlorophenol in the solidified wood preservation waste soil. Cadmium was near or below detection in the leachates from both the equilibrium extraction and the dynamic leach test, as were arsenic, lead, and thallium for the solidified aluminum coil plating sludge samples. Thus, the concentrations measured in the dynamic leach test leachates did not approach the equilibrium concentration unless the equilibrium concentration was very near or below detection.

5.3 Micromorphological Evaluation Results

The results from X-ray diffraction, scanning electron microscopy, and optical microscopy are very qualitative; thus, it was not possible to conduct a statistical analysis of the results from these techniques. The results from energy dispersive X-ray analysis are only semiquantitative on a macroscopic scale because of sample heterogeneity, and even on a microscopic scale because a number of parameters affecting the X-rays emerging from the sample were not well known. Consequently, a statistical analysis was not carried out for the energy dispersive X-ray analysis results either.

The features of the solidified products of the various waste types that were observed using each of the micromorphological characterization techniques are summarized in the following sections. For each method, the observations for each solidified product were compared with the observations made for the untreated wastes and for common solidification additives, such as portland cement and fly ash (without the addition of a waste component). The results from each technique are most useful when interpreted in conjunction with the results of the other techniques.

5.3.1 X-ray Diffraction

This technique was conducted using two sources of monochromatic X-rays: copper and molybdenum. Copper is usually the preferred source, as it results in more distinct, widely spaced peaks and a lower background pattern. It was used for X-ray diffraction analysis of the Wastewater Technology Centre synthetic solution solidified products, and some wood preservation waste soil solidified products. Because of laboratory limitations, molybdenum was used as an X-ray source for X-ray diffraction analysis of the Waterways Experiment Station synthetic sludge solidified products, and the remaining wood preservation waste soil solidified products.

The X-ray diffraction patterns were compared with known patterns to identify the crystalline phases present in the samples. The sample matrices were complex, resulting in a number of unidentified peaks in most X-ray diffraction patterns. Also, peaks were often shifted slightly with respect to the pure compounds because of impurities. It was found that the peaks for 10 phases were identified most frequently: portlandite, ettringite, calcium aluminum hydrate, dicalcium silicate, tricalcium silicate, calcite, gypsum, quartz, mullite, and maghemite. Table 31 summarizes the crystalline phases identified for each solidified product. In addition to the most common phases listed, other phases identified included opal-ct, montmorillonite, magnetite, lead sulphate, monosulphate, and anhydrite.

Portlandite ($Ca(OH)_2$), ettringite ($CaSO_4 \cdot 3(3CaO \cdot Al_2O_3) \cdot 32H_2O$), monosulphate (which can be a more stable form of ettringite, or is formed when insufficient gypsum is present), calcium aluminum hydrate, and gypsum ($CaSO_4 \cdot 2H_2O$) are crystalline phases present in hydrated portland cement. Figure 4 shows a typical X-ray diffraction pattern for portland cement using copper X-ray sources (molybdenum patterns show a distinctive "portland cement hump" background, caused by the relatively low crystallinity of the sample). The above-mentioned phases have been labelled. Figure 5 shows an example of an X-ray diffraction pattern for a solidified product in which the phases typical of portland cement can be identified.

The presence of portlandite in solidified samples may also be attributed to the addition of excess lime, separately from portland cement. $CaSO_4$ (as anhydrite or gypsum) is also a common solidification additive, either separately or in conjunction with portland cement. Dicalcium and tricalcium silicates ($2CaO \cdot SiO_2$ and $3CaO \cdot SiO_2$) are found in unhydrated portland cement. Their presence in the X-ray diffraction pattern for a solidified product indicates that complete hydration has not taken place, either because of insufficient water addition or some retarding mechanism.

Calcite ($CaCO_3$) was identified frequently, particularly in the Wastewater Technology Centre synthetic solution solidified products. This phase may be a product of carbonation of portlandite when it comes in contact with air; however, the amounts observed in the samples were unusually high.

Maghemite and magnetite (two forms of Fe_2O_3) are characteristic phases found in fly ash. Mullite ($3Al_2O_3 \cdot 2SiO_2$) is found in type F, rather than type C, fly ash. Figures 6 and 7 show typical X-ray diffraction patterns with labelled peaks for type C fly ash and type F fly ash respectively. Quartz is invariably present in fly ash, as well as other minerals. Often, the presence of fly ash phases (particularly mullite) in the X-ray diffraction pattern for a solidified product will be accompanied by an absence of portlandite peaks, since excess fly ash will consume free lime. An example of an X-ray diffraction pattern for a solidified waste apparently containing fly ash is presented in Figure 8.

In addition to the peaks caused by solidification additives, the X-ray diffraction patterns showed evidence of the presence of the waste. The pattern for one of the solidified Wastewater Technology Centre

Table 31 X-ray Diffraction Observations

(a) WTC synthetic solution solidified products

Industrial Participant ID Code	Portlandite	Ettringite	CAH	C_2S,C_3S	Calcite	Gypsum	Quartz	Mullite	Maghemite	Other
AAKC	x	x			x	12 600				
BABI	x	x	x	x	x					Diatom (opal-ct,600)
BAHE				x	x	x				Montmorillonite(1400) Magnetite
DEAI	?		x		1 400(?)	x				
DEIF	1 600		x	x	x					
DGBF	?	x		x	x		2 900			
EAFI	x	x	x	x	3 000		x			
EEBH		x			3 800		x	x	x	
EFBB	x	x	x	x	3 000	x	x	x	x	
FAIG			x		2 300		x	x		
FCCI	3 200	x	x		x					
FFKK	1 000	x	x	x	x		x			
HDGF	?	x			x	x	1 900	x		Monosulphate
IGIF	x		x			x	x			

(b) WES synthetic sludge solidified products

Industrial Participant ID Code	Portlandite	Ettringite	CAH	C_2S,C_3S	Calcite	Gypsum	Quartz	Mullite	Maghemite	Sludge	Other
AIEI	x	1 000					x				
AIKC	x	?					x		x	2 300	
BBIE	x	x	x	x			x			1 700	
BDGE	x					x				2 900	
BEDF	x			x		x	x	x	x	1 300	
DABB	?		x	x		x		x	x	1 500	
EAHK	x	x				x	x	x		1 500	
ECGH	x	x			x	x	900	?		?	
GBAB	1 400	x		x		x		x	x	x	
GBFB	x	x		x			2 700		?		
HGAB	1 800	x		x	?	x		x			Magnetite
KEAH	?	x		x		x	x		x	2 100	
KHEF	1 600	x	x	x						x	
KIAK	500	x		x						x	

(c) Wood preservation waste soil solidified products

Industrial Participant ID Code	Portlandite	Ettringite	CAH	C_2S,C_3S	Calcite	Gypsum	Quartz	Mullite	Maghemite	Other
ADDI				x	x	?	1 600			
BIDA	x	x	x		x		3 300	?		
BKDE	x	?			x		3 500	x	x	
CDHB	x	x		x	x	x	2 400			Anhydrite?
DEDB							6 300			Anhydrite?
DIDG						x	4 600			Anhydrite
EIDA		?					800			
FDGI	x	x			x		4 700	x		
HBEG							6 400			
HIKH		?				?	5 000	?		
IAHE		x				x	3 300	?		
KBKC			x	x		x	800			
KGAH							1 300			

Note: For each solidified product, the peak height of the most intense peak is given in counts per second (cps). The main peak height is measured above the background; for the solidified products in (a), (b), and (c), the backgrounds are approximately 200, 700, and 700 cps respectively.
CAH, calcium aluminum hydrate.
C_2S and C_3S, dicalcium silicate and tricalcium silicate.
x, this phase was identified in the X-ray diffraction pattern.
?, this phase was tentatively identified in the X-ray diffraction pattern.

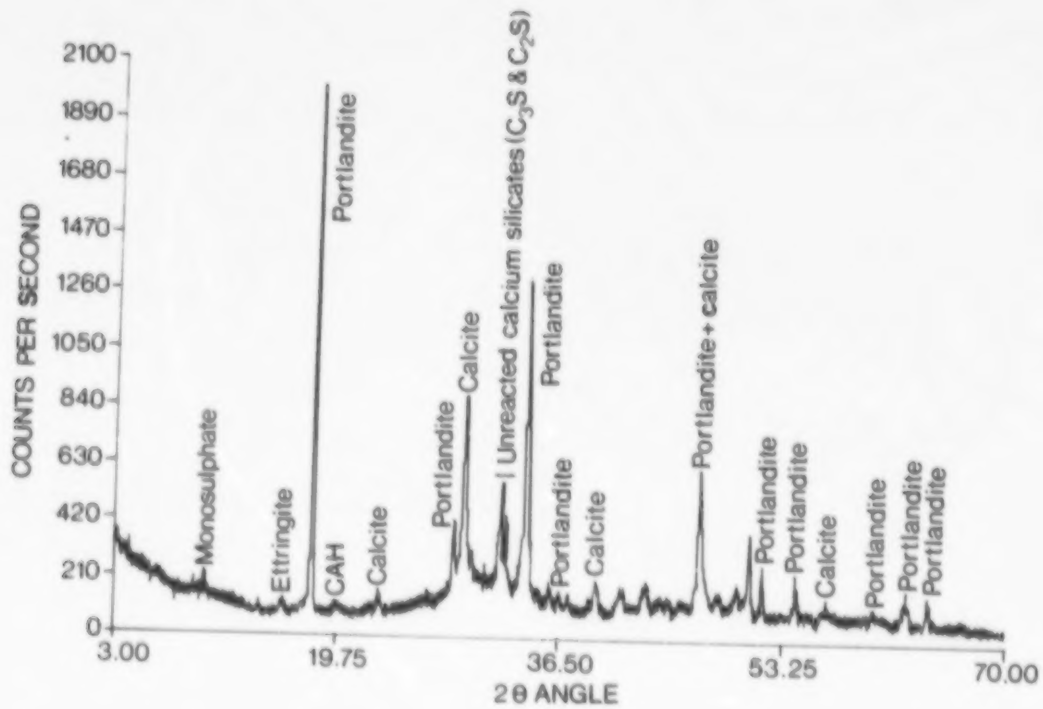

Figure 4 Typical X-ray Diffraction Pattern for Portland Cement

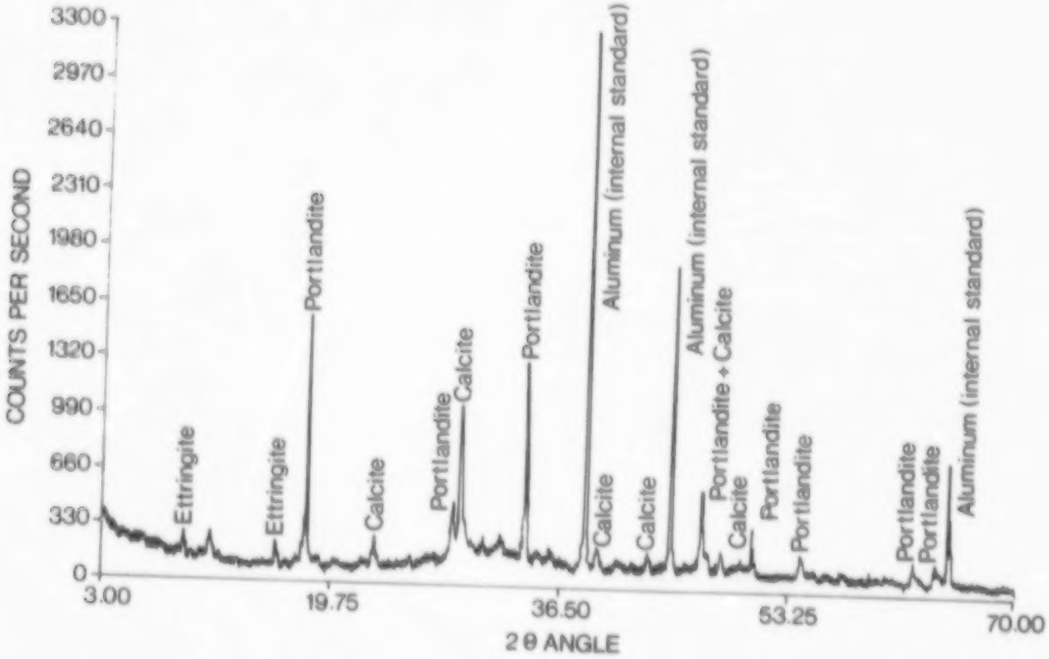

Figure 5 Example of X-ray Diffraction for a Solidified Product With Portland Cement

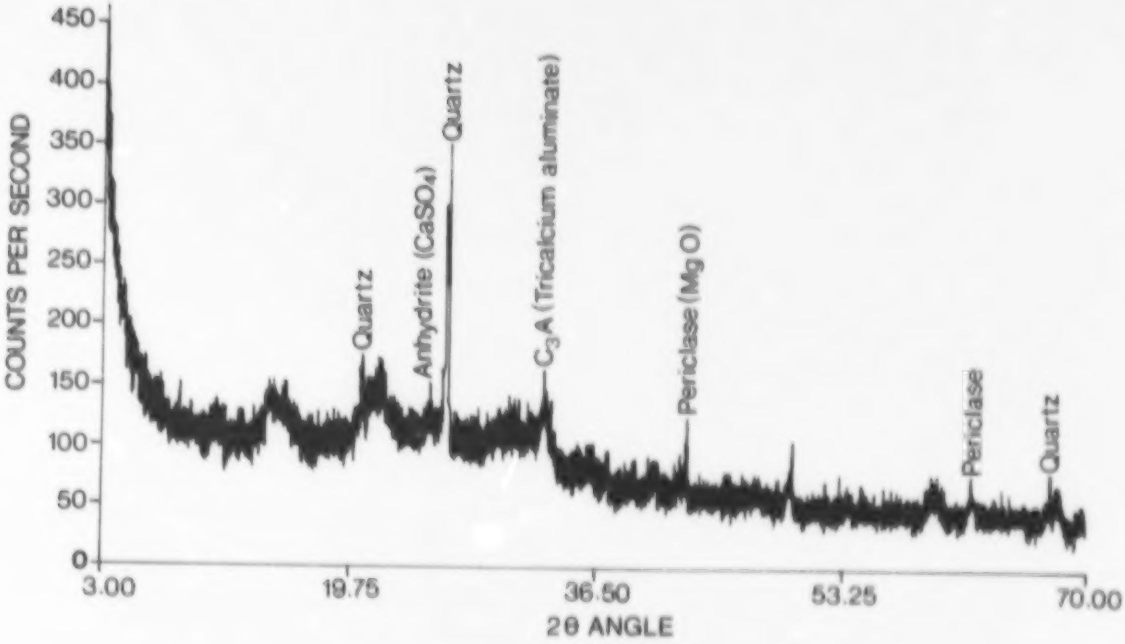

Figure 6 **Typical X-ray Diffraction Pattern for Type C Fly Ash**

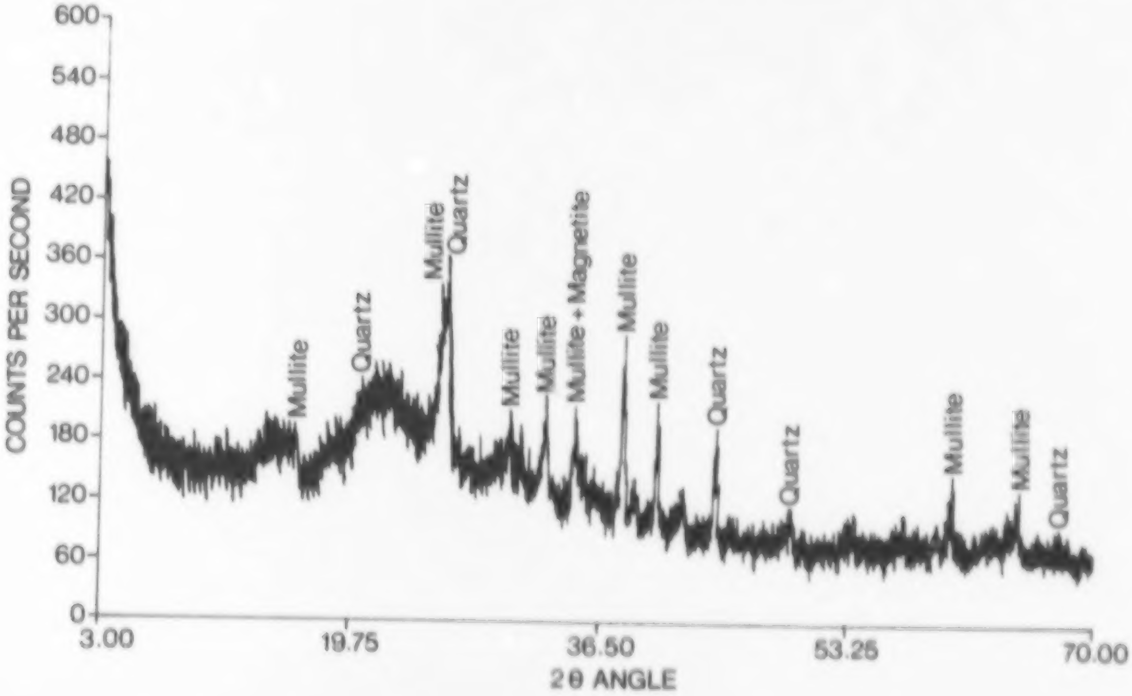

Figure 7 **Typical X-ray Diffraction Pattern for Type F Fly Ash**

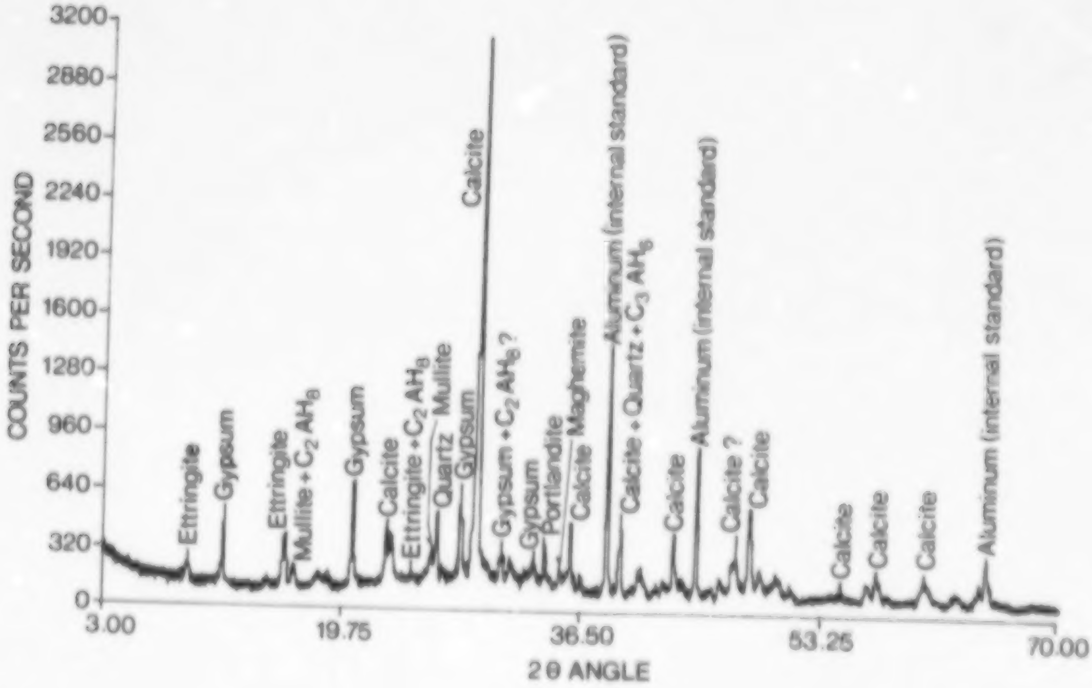

Figure 8 **Example of an X-ray Diffraction Pattern for a Solidified Product Containing Fly Ash**

synthetic solution products (Figure 9) showed a series of peaks indicative of an interaction between phenol and cement (Roy et al., 1989). The Waterways Experiment Station synthetic sludge solidified products, and the wood preservation waste soil solidified products, often showed peaks contributed by the original waste matrix.

Figure 10 shows the X-ray diffraction pattern for the pure Waterways Experiment Station synthetic sludge. Strong, distinct peaks against a low background indicate that the sludge is highly crystalline. Calcite is clearly one of the components of the sludge, but many of the distinctive sludge peaks remain unidentified. It is likely that they are caused by mixtures of the different metal hydroxides. The characteristic sludge peaks can easily be distinguished in the X-ray diffraction patterns of 11 of the 14 solidified sludge samples, and tentatively identified in two others. An example is shown in Figure 11.

Figure 12 shows an X-ray diffraction pattern for the untreated wood preservation waste soil, with predominant quartz peaks and other unidentified peaks. This waste matrix strongly influenced the X-ray diffraction patterns for the wood preservation waste soil solidified samples. In every case, the quartz particles in the soil dominated the X-ray diffraction pattern. The complex nature of the matrix made identifying any other phases very difficult.

5.3.2 Scanning Electron Microscopy

The sample features observed using scanning electron microscopy (SEM) are listed in Table 32 for each of the solidified products examined. In the table, a plate number is given for some observations, which may be referred to for an illustration of the feature under discussion. Photomicrographs for every solidified sample studied using scanning electron microscopy have not been reproduced in this report to avoid unnecessary repetition.

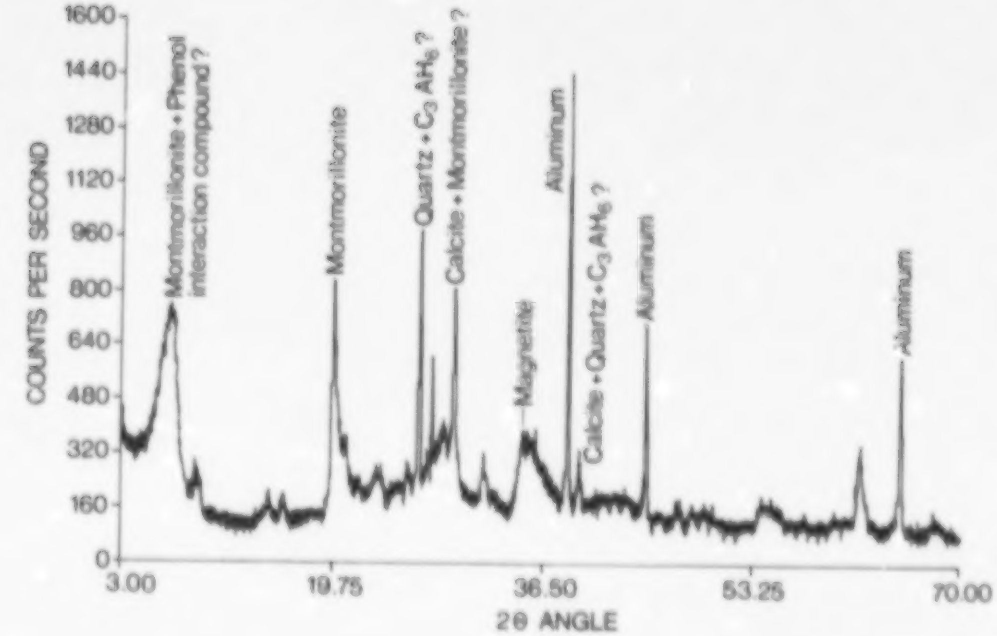

Figure 9 Example of an X-ray Diffraction Pattern Showing the Interaction Between Phenol and the Matrix

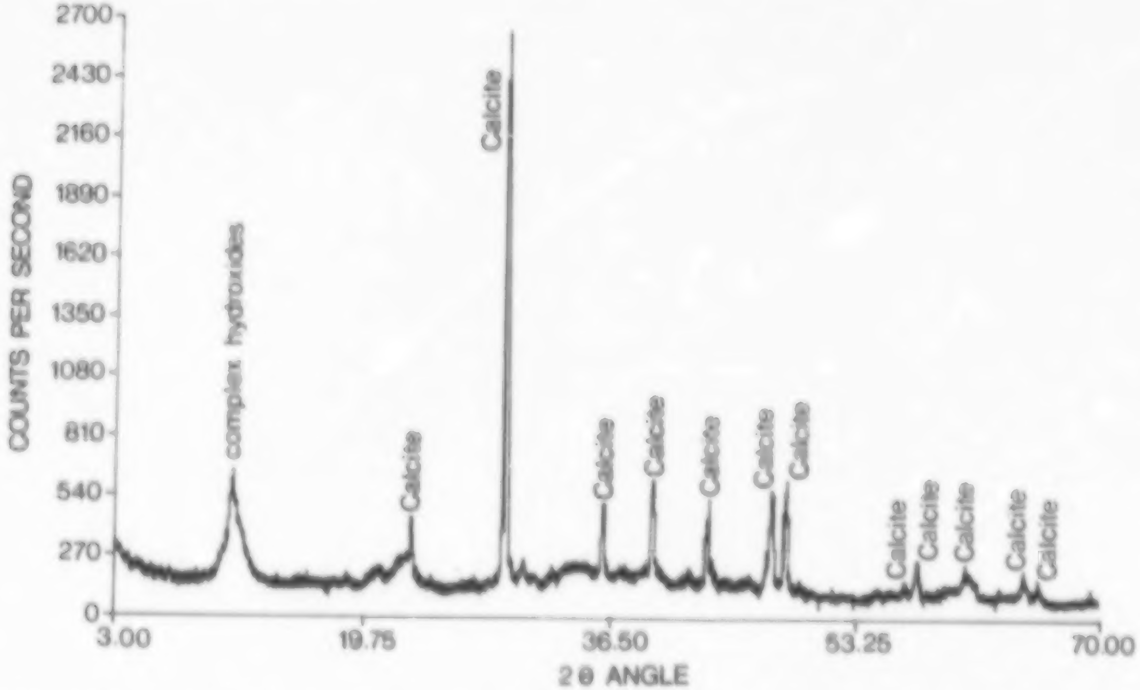

Figure 10 X-ray Diffraction Pattern for the Waterways Experiment Station Synthetic Sludge

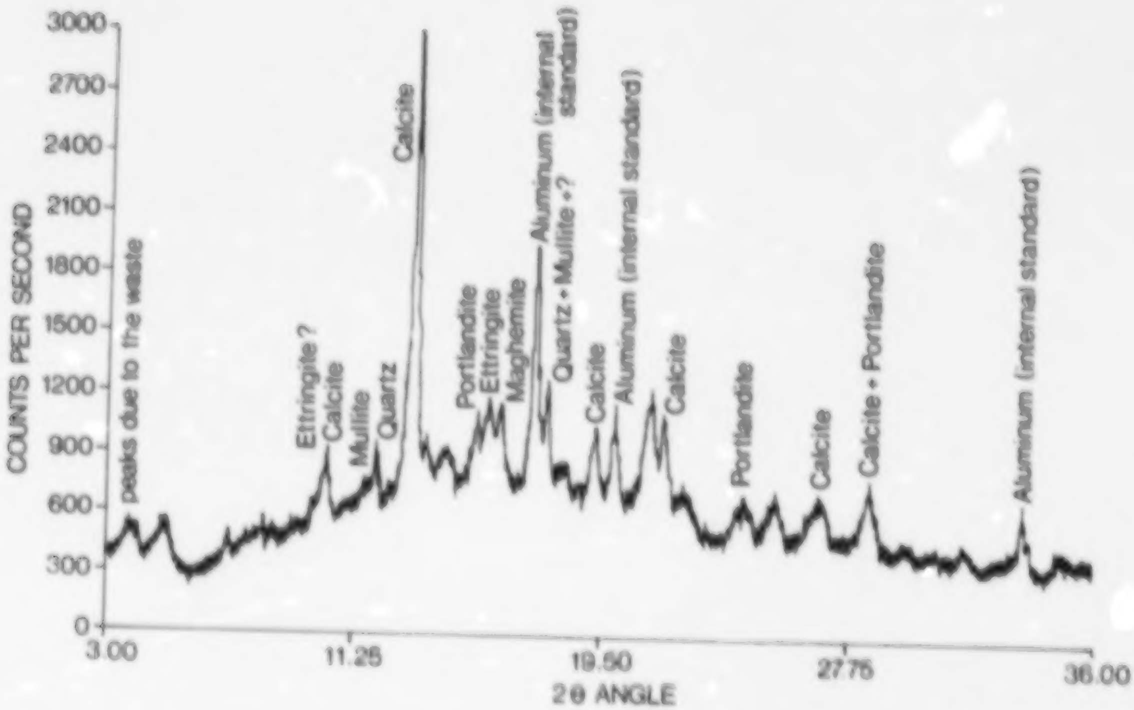

Figure 11 Example of an X-ray Diffraction Pattern for a Solidified Product Containing the Waterways Experiment Station Sludge

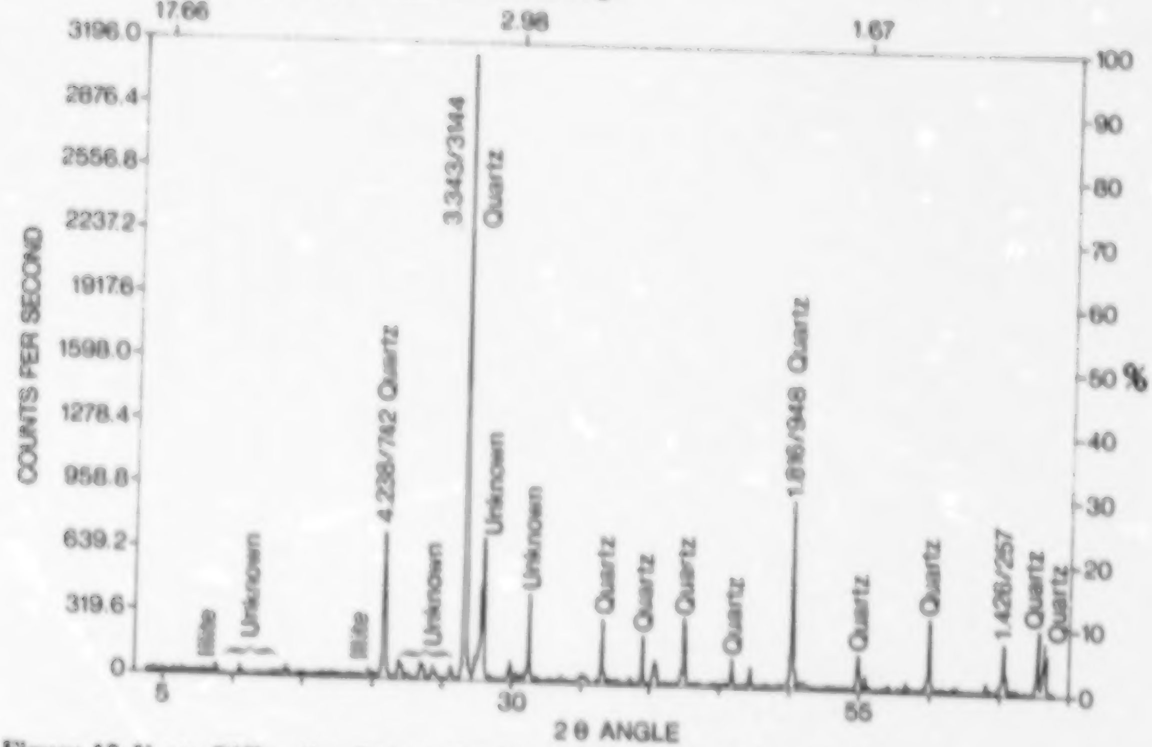

Figure 12 X-ray Diffraction Pattern for Wood Preservation Waste Soil

Table 32 Scanning Electron Microscopy Observations

(a) WTC synthetic solution solidified products

Industrial Participant ID Code	Morphological Observations	Plate No.	Magnification
AAKC	Low porosity		
	Very homogeneous with occasional very smooth areas		
	Morphology unlike ordinary portland cement		
	Higher magnifications show a layered platelike structure		
BABI	Moderate interparticle porosity		
	Homogeneous		
	Abundant fly ash, largely unconsumed by reactions	6	×300
	Abundant fossilized diatom frustules	10	×4 000
BAHE	Large crevice-like voids		
	Heterogeneous		
	Platelike crystals		
	Needlelike crystals	1	×5 000
DEAI	Large voids		
	Homogeneous		
	Glassy honeycomb-like structure	9	×20 000
DEIF	No SEM was conducted for this sample		
DGBF	Very low porosity		
	Heterogeneous		
	Late-forming radiating platelike crystals		
	Needlelike crystals in small interparticle voids		
	Weathered quartz grains		
	Platelike crystals		
	Other less distinct crystals		
EAFI	Moderate porosity		
	Some voids caused by hollow fly ash particles		
	Platelike crystals		
	Other less distinct crystals		
	Inclusion composed of dense fine crystals		
EEBH	Low porosity		
	Abundant fly ash		
	Some fly ash is partially reacted		
	Long flat crystals		
	Very dense areas, like CSH in portland cement		
EFBB	Large voids between cluster of different phases		
	Reacted fly ash particles		
	Elongated crystals with pyramidal faces		
	Needlelike crystals		
	Bulk of sample is cryptocrystalline		
FAIG	Low porosity		
	Largely consumed fly ash grains		
	Scattered platelike crystals		
	Larger books of hexagonal platelike crystals	2 3	×1 960 ×6 000
FCCI	Extremely dense structure		
	Spherical voids from trapped air bubbles		
	Needlelike crystals		
	Flat elongated crystals		
	Some platelike crystals		
FFKK	Moderate porosity - some fly ash, partly consumed by reactions		
	Bulk of sample is cryptocrystalline		
HDGF	Moderate porosity		
	Abundant fly ash		
	Platelike crystals		
	Bulk of sample has scalclike CSH structure	5	×5 000
IGIF	Moderate porosity		
	Heterogeneous		
	Needlelike crystals		
	Bulk of samples has scalelike CSH structure		
	Inclusion composed of dense fine crystals		

Table 32 Scanning Electron Microscopy Observations (cont'd)

(b) WES synthetic sludge solidified products

Industrial Participant ID Code	Morphological Observations	Plate No.	Magnification
AIEI	Low porosity Homogeneous Hexagonal platelike crystals Needlelike crystals Inclusion composed of smaller particles		
AIKC	Large, crevice-like voids Cryptocrystalline		
BBIE	Very low porosity Homogeneous Platelike crystals in voids		
BDGE	Elongated crystals with pyramidal faces		
BEDF	Very low porosity Homogeneous Fly ash spheres Some hexagonal platelike crystals Some thin, elongated crystals Bulk of sample has scalelike CSH structure		
DABB	Moderate porosity Homogeneous Occasional thin, elongated crystals		
EAHK	Extremely dense structure with occasional spherical voids Evidence of consumed fly ash grains Platelike crystals Needlelike crystals		
ECGH	Low porosity Some fly ash particles, partly consumed by reactions Hexagonal platelike crystals Encapsulated sludge particle	12	×500
GBAB	Low porosity Homogeneous Fly ash particles, partly consumed by reaction Platelike crystals	7	×6 800
GBFB	Low porosity Homogeneous Some thick hexagonal platelike crystals Some needlelike crystals		
HGAB	Low porosity Some voids caused by hollow fly ash particles Some fly ash particles, almost completely consumed by reactions Abundant needlelike crystals		
KEAH	Low porosity Homogeneous Elongated rectangular crystals		
KHEF	Moderate porosity Cryptocrystalline		
KIAK	Low porosity Ellipsoidal sludge grains embedded in a finer grained matrix	13	×500

Two types of porosity were observed using scanning electron microscopy: 1) interparticle porosity composed of pores of different sizes between the small crystals and particles that form the matrix, and 2) a larger scale porosity caused by holes and voids within, and cracks between, denser areas. There was no apparent relationship between these observations and the calculated porosity presented in Section 5.1.4. Many of the phases identified in the X-ray diffraction patterns were also observed using scanning electron microscopy. Needlelike crystals, typical of ettringite (Plate 1), and hexagonal platelike crystals, typical of portlandite, monosulphate, and calcium aluminum hydrate (Plates 2 and 3),

Table 32 Scanning Electron Microscopy Observations (cont'd)

(c) Wood preservation waste soil solidified products

Industrial Participant ID Code	Morphological Observations	Plate No.	Magnification
ADDI	No SEM was conducted for this sample		
BIDA	Very low porosity Homogeneous Some needlelike crystals Scalelike CSH		
BKDE	Large voids Homogeneous Quartz grains		
CDHB	Low porosity Wood splinters Uniformly distributed quartz particles	15 16	×1 000 ×3 000
DEDB	Uniformly distributed quartz particles Some cubic crystals		
DIDG	Low porosity Homogeneous Abundant thin, elongated crystals	8	×1 000
EIDA	No SEM was conducted for this sample		
FDGI	No SEM was conducted for this sample		
HBEG	Low porosity Cryptocrystalline		
HIKH	Low porosity Homogeneous Melting of smooth oblate particles under the electron beam		
IAHE	Very low porosity Homogeneous Some fly ash particles		
KBKC	Low porosity Some spherical voids Fly ash particles		
KGAH	No SEM was conducted for this sample		

were seen with great frequency. However, although evidence of the phases present in portland cement was encountered, the overall morphology of most of the samples was unlike that of portland cement (Plate 4). The calcium silicate hydrate (CSH) often seen in the solidified samples usually showed a dense, heavy scalelike structure (Plate 5), whereas in ordinary portland cement it appears in many forms, including the fingerlike structure shown in Plate 4.

Fly ash particles have a very characteristic morphology, which was seen in many of the solidified products observed using scanning electron microscopy. As shown in Plate 6, unreacted fly ash appears as spherical particles scattered throughout the matrix. Reaction products may often be seen on the surface of the fly ash (Plate 7), or the fly ash may be completely consumed by reactions. Uncommon features observed included gypsum crystals (Plate 8), a glassy honeycomb-like structure (Plate 9), and diatom skeletons (Plate 10). The observation of fossilized diatom frustules corresponded with the finding of opal-ct, a form of SiO_2, using X-ray diffraction. Various unidentifiable crystals were also noted.

Plate 11a, b, and c shows scanning electron microscopy photomicrographs of the dried, unsolidified Waterways Experiment Station synthetic sludge. It is apparent that this waste is highly crystalline, and that there are at least three distinctly different crystal morphologies. The presence of sludge particles, encapsulated in the matrix, may be

Plate 1 Needlelike Crystals Typical of Ettringite (SEM, solidified product BAHE, ×5000)

Plate 2 Platelike Crystals Typical of Portlandite, Monosulphate, and Calcium Aluminum Hydrate (SEM, solidified product FAIG, ×1960)

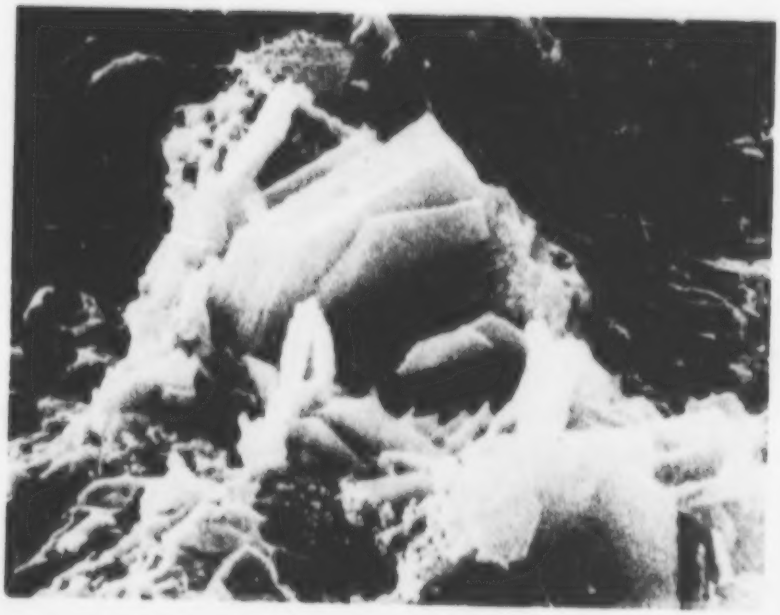

Plate 3 Platelike Crystals Typical of Portlandite, Monosulphate, and Calcium Aluminum Hydrate (SEM, solidified product FAIG, ×6000)

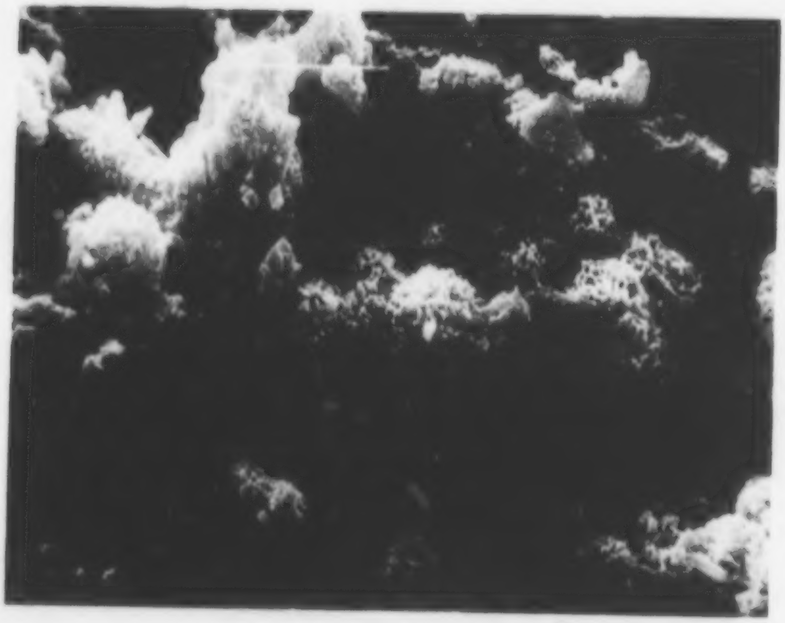

Plate 4 Hydrated Portland Cement Showing Fingerlike Calcium Silicate Hydrate (SEM, ×2500)

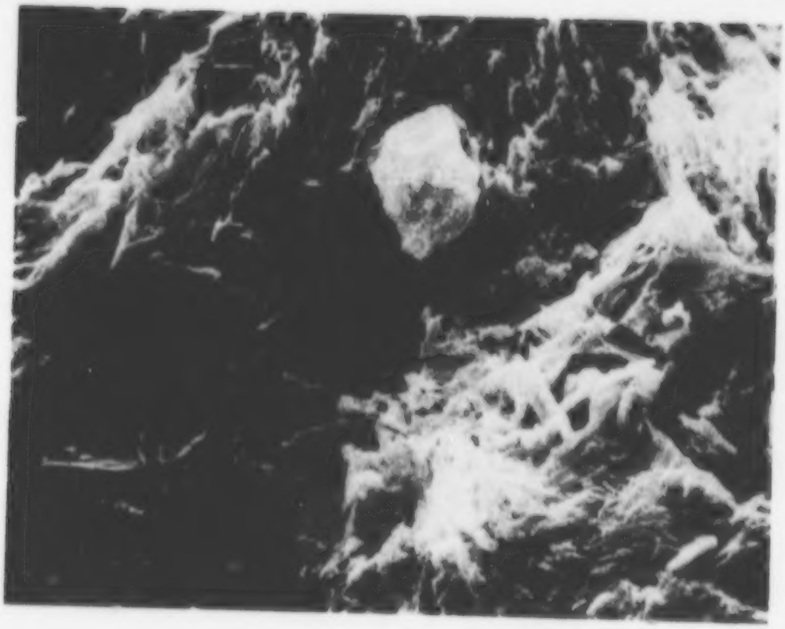

Plate 5 Dense, Scalelike Calcium Silicate Hydrate (SEM, solidified product HDGF, ×5000)

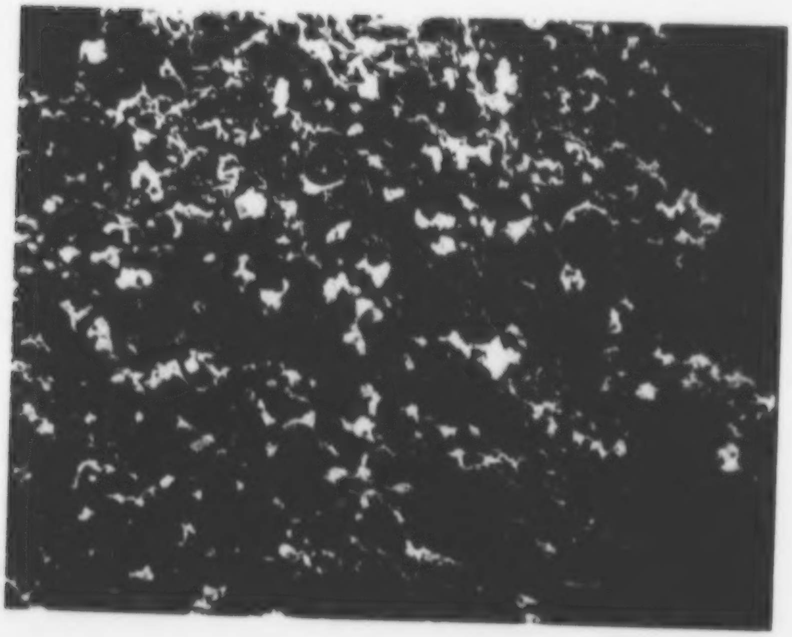

Plate 6 Spherical Fly Ash Particles (SEM, solidified product BABI, ×300)

Plate 7 Reaction Products on the Surface of Fly Ash (SEM, solidified product GBAB, ×6800)

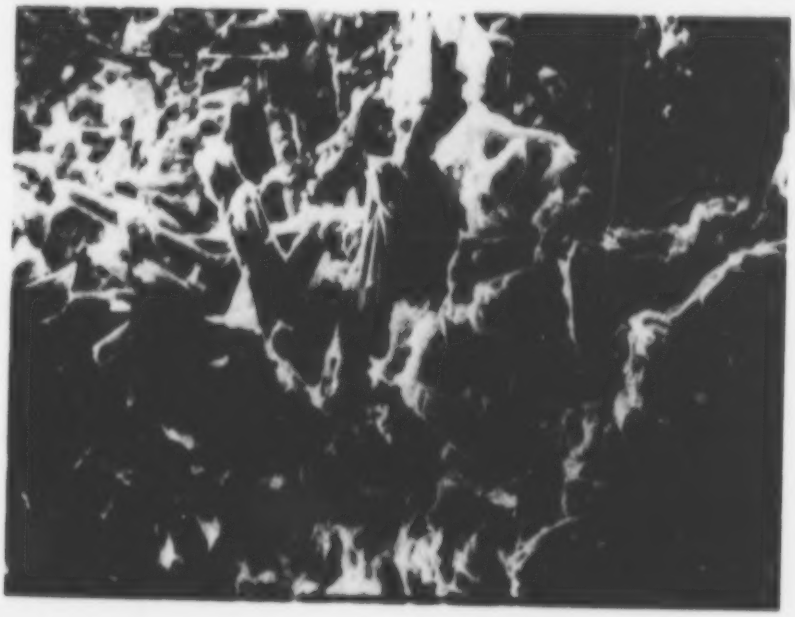

Plate 8 Calcium Sulphate Crystals (SEM, solidified product DIDG, ×1000)

Plate 9 Glassy, Honeycomb-like Structure (SEM, solidified product DEAI, ×20 000)

Plate 10 Fossilized Diatom Frustules (SEM, solidified product BABI, ×4000)

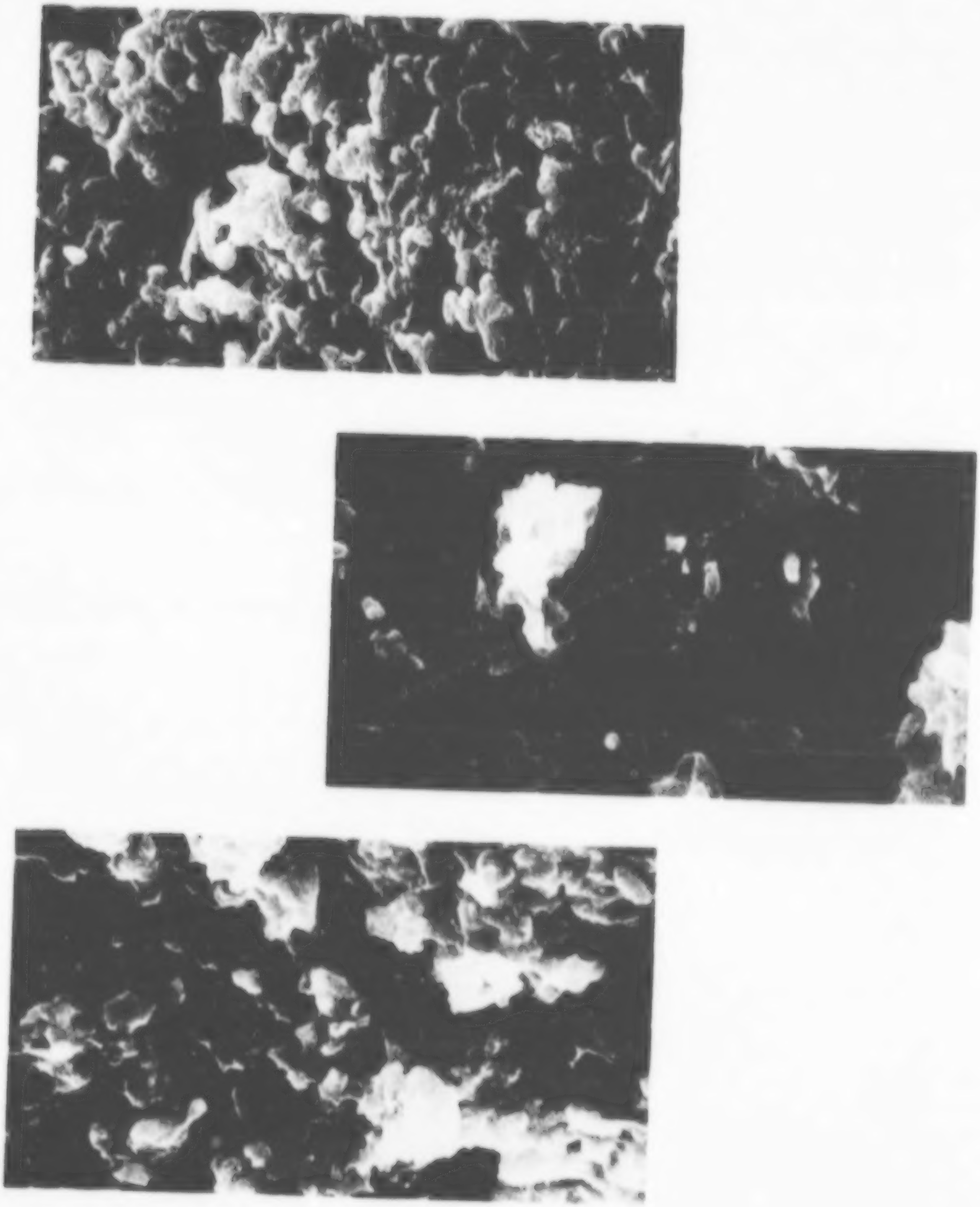

Plate 11 Waterways Experiment Station Synthetic Sludge (SEM, ×5000)

observed for many of the Waterways Experiment Station synthetic sludge solidified products, as demonstrated in Plates 12 and 13.

Plate 14 shows a photomicrograph of the raw wood preservation waste soil. The sample consists of large quartz grains coated with fine-grained material. For the wood preservation waste soil solidified products, the soil matrix dominated the scanning electron microscopy photomicrographs, as well as the X-ray diffraction patterns. Wood fragments (Plate 15) and quartz grains (Plate 16) were ubiquitous. However, one sample in particular was interesting in that the electron beam caused melting of the sample surface. Four of the 13 solidified products for this waste could not be examined using scanning electron microscopy because of an accumulation of electrons on the sample surface due to an inadequate gold coating on the sample. The poor quality of the coating was caused by the presence of a high concentration of volatile compounds (including organic compounds and water) in the sample.

5.3.3 Energy Dispersive X-ray Analysis

Energy dispersive X-ray analysis (EDXRA) was conducted for many of the notable features observed using scanning electron microscopy. Table 33 summarizes the analyses carried out. As was the case for the scanning electron microscopy photomicrographs, the spectrum for every analysis conducted has not been included in this report.

The use of cementitious and pozzolanic binders resulted in the appearance of strong calcium, silicon, and aluminum peaks. Sulphur, iron, and magnesium also appear. The ratios of these components can be used to draw inferences concerning the composition of the matrix. For example, compared with cement, most pozzolans have a very high ratio of calcium to silicon and aluminum; a relatively low ratio of calcium to sulphur is an indication that gypsum has been added.

The most useful aspect of energy dispersive X-ray analysis is the opportunity it provides to examine the distribution of the

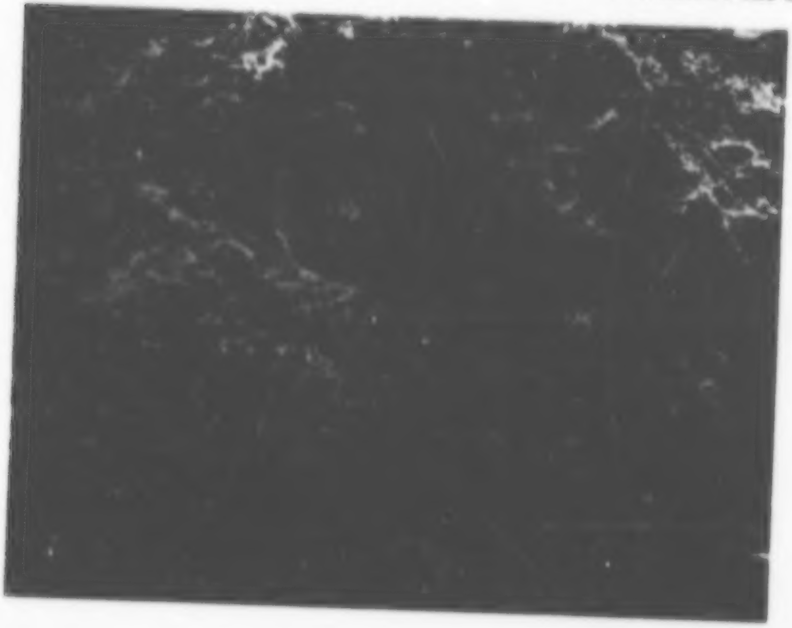

Plate 12 Encapsulated Sludge Particle (SEM, solidified product ECGH, ×500)

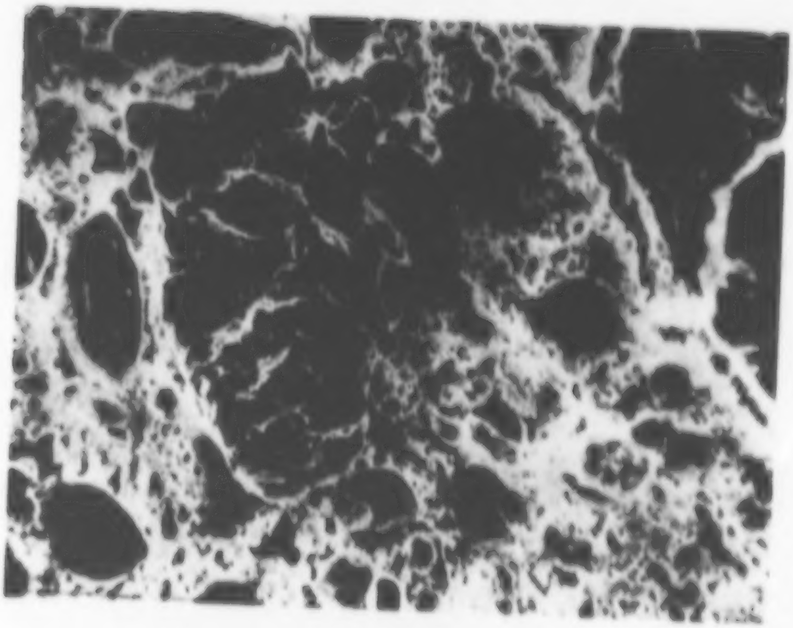

Plate 13 Numerous Encapsulated Sludge Particles (SEM, solidified product KIAK, ×500)

Plate 14 Wood Preservation Waste Soil (SEM, ×375)

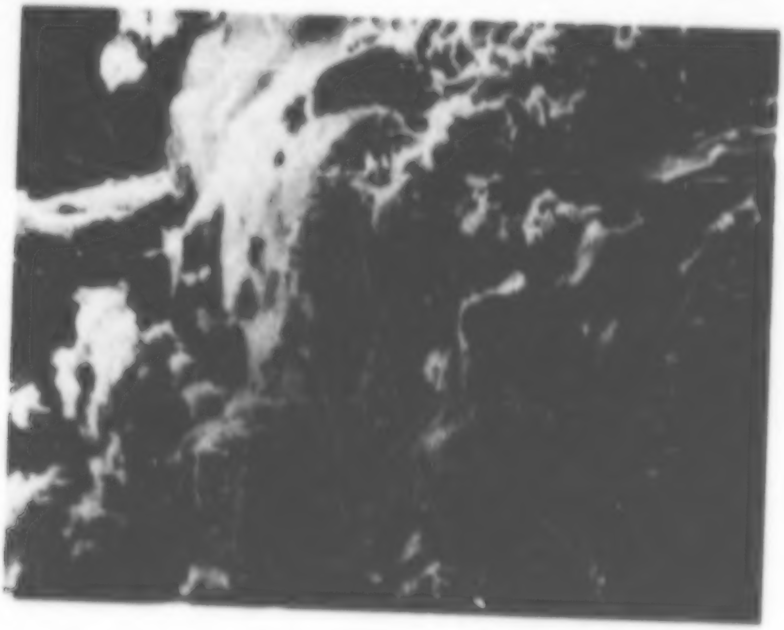

Plate 15 Wood Fibre (SEM, solidified product CDHB, ×1000)

Plate 16 Quartz Grain (SEM, solidified product CDHB, ×3000)

Table 33 **Energy Dispersive X-ray Analyses**

(a) WTC synthetic solution solidified products

Industrial Participant ID Code	Description of Analysis Location	Other Peaks
AAKC	Flat layered particle	Cd
	Average of 0.05 mm diameter area	Cd,Cr
BABI	Fly ash sphere	Pb,Cl,Ti
	Diatom skeleton	Pb,Cl,Cd
	Average of 0.8 mm diameter area	Pb,Cl,Cd
BAHE	Irregularly shaped grain	Cd,Ti,Cr,Mn
	Average of 1 mm diameter area	
DEAI	Spherical particle	Cl,Cd,Cr
	Particle with sharp, angular faces	
DEIF	No EDXRA was conducted for this sample	
DGBF	Average of 1.4 mm diameter area	
EAFI	Average of 0.6 mm diameter area	Cl,K
	Large grain	Cl,K
EEBH	Small round grain	Cl,K
	Crystalline grain	Cl,K
	Elongated crystalline grain	Cl,K
EFBB	Ellipsoidal grain	Cl.Cd,K
	Elongated rectangular particle	Cl,Cd,K,Cr
	Well-defined crystal	
FAIG	Smooth rounded grain	Pb,Cl,Cd,K
FCCI	Particle with angular faces	K
FFKK	Reacted fly ash sphere	K
HDGF	Average of 1 mm diameter area	Cl,Ti
IGIF	Fine-grained binder material	K
	Oblate particle	Cl,Cd,K,Cr

contaminants in the solidified waste matrix. Spot analyses of different phases will show whether a contaminant is homogeneously distributed or associated with a particular phase. However, no conclusion regarding the environmental stability of the contaminant immobilization mechanism can be reached using this method alone.

For the solidified Wastewater Technology Centre synthetic solution samples, the only contaminant easily distinguished by energy

Table 33 **Energy Dispersive X-ray Analyses (cont'd)**

(b) WES synthetic sludge solidified products

Industrial Participant ID Code	Description of Analysis Location	Other Peaks
AIEI	Layered platelike crystal	
	Layered platelike crystal	Cr,Ni
AIKC	No EDXRA was conducted for this sample	
BBIE	No EDXRA was conducted for this sample	
BDGE	Elongated crystal with pyramidal faces	Cr,Ni
BEDF	No EDXRA was conducted for this sample	
DABB	No EDXRA was conducted for this sample	
EAHK	Bulk matrix	Cr,Ni,Na,K
	Bulk matrix	Cr,Ni,Na,K
ECGH	Encapsulated sludge particle	Cr,Ni
GBAB	Encapsulated sludge particle	Cr,Ni
	Surface of fly ash particle	Cr,Ni
GBFB	Poorly crystalline material	Cr,Ni
HGAB	No EDXRA was conducted for this sample	
KEAH	Elongated rectangular crystals	Cr,Ni
	Amorphous calcium silicate hydrate	Cr,Ni
KHEF	No EDXRA was conducted for this sample	
KIAK	Encapsulated sludge particle	Cd,Cr,Ni
	Matrix surrounding sludge particles	Cd,Cr,Ni

dispersive X-ray analysis was chromium. Unfortunately, in the energy dispersive X-ray analysis spectra, the arsenic peak overlaps with the magnesium peak, the lead peak overlaps with the sulphur peak, and the cadmium peak overlaps with the potassium peak, whereas phenol cannot be detected. Even chromium was not seen to any great degree in any of the samples. This may indicate homogeneous distribution of the contaminants in the matrix, rather than concentration in any particular phase. Chlorine (from the dissolution of $CrCl_3$ in the raw waste solution) was apparent in the majority of the spectra.

For the Waterways Experiment Station synthetic sludge solidified products, only chromium and nickel could be detected by energy dispersive X-ray analysis. Mercury concentrations were too low, and the cadmium peak overlapped with the potassium

Table 33 Energy Dispersive X-ray Analyses (cont'd)

(c) Wood preservation waste soil solidified products

Industrial Participant ID Code	Description of Analysis Location	Other Peaks
ADDI	No EDXRA was conducted for this sample	
BIDA	No EDXRA was conducted for this sample	
BKDE	Large irregularly shaped particle	Cl,K
CDHB	Crystalline grain	Na,K
	Bulk matrix - layered particle	Cl,K
	Flat particle	Cl,K
DEDB	Bulk matrix	Na,K,Cl
	Bulk matrix	Na,K,Cl
DIDG	Rectangular crystal	Cl
	Gypsum crystal	
EIDA	No EDXRA was conducted for this sample	
FDGI	No EDXRA was conducted for this sample	
HBEG	No EDXRA was conducted for this sample	
HIKH		Cl,K
		Cl
IAHE	Fly ash particle	Cl,K
	Rectangular particle or crystal	Cl,K
KBKC	Fly ash particle	Cl,K
KGAH	No EDXRA was conducted for this sample	

peak in the spectra. The grains identified by scanning electron microscopy as sludge particles encapsulated in the matrix were subjected to energy dispersive X-ray analysis. Comparison of the spectra from these grains with the spectrum of the pure sludge showed that their composition was identical. However, analysis of other phases in the surrounding matrix showed that Cr and Ni were present in these as well. This phenomenon, which was observed to varying extents in the different solidified products, indicates that some respeciation of the contaminants from the sludge into the matrix has taken place.

Six of the 13 wood preservation waste soil solidified products could not be analyzed using this technique because of charging caused by a poor carbon coating on the sample. Chlorine (in this case an indicator for the location of pentachlorophenol) appeared

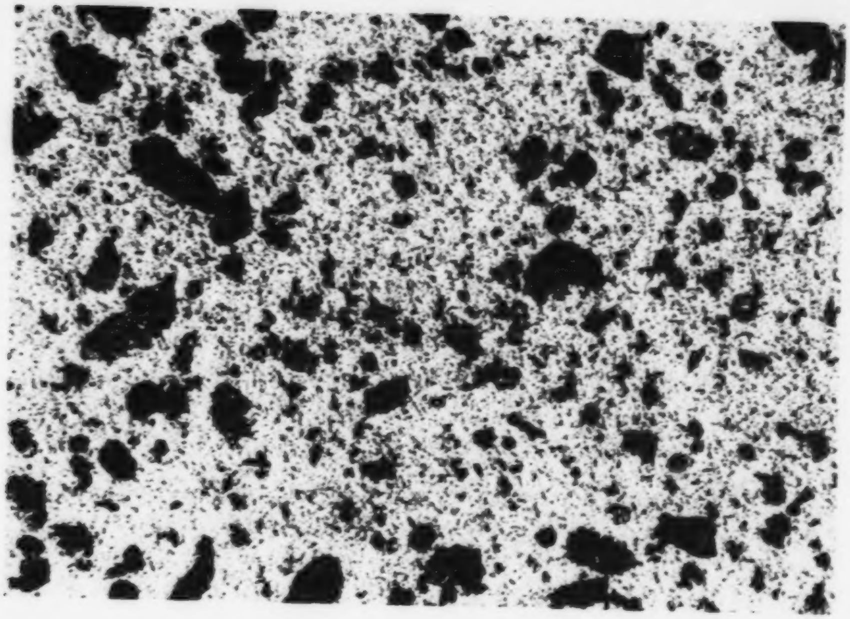

Plate 17 Optical Microscopy Showing Opaque Particles (solidified product GBAB, ×40)

Plate 18 Optical Microscopy Showing Opaque Particles in the Waterways Experiment Station Synthetic Sludge (×100)

to be distributed homogeneously in some matrices, and heterogeneously in others.

The presence of other peaks, such as strong sodium, potassium, and chlorine peaks in one of the Waterways Experiment Station synthetic sludge solidified products, indicates the addition of a solidification additive containing these elements.

5.3.4 Optical Microscopy

Examination of several of the Waterways Experiment Station synthetic sludge solidified products under the optical microscope showed numerous opaque particles amongst and within more translucent particles (Plate 17). These opaque particles could not be seen in pure cement. However, examination of the untreated Waterways Experiment Station synthetic sludge (Plate 18) showed that it was composed entirely of opaque particles; thus, it appears that the Waterways Experiment Station synthetic sludge particles are physically encapsulated in the solidified waste matrix.

Examination of the solidified Wastewater Technology Centre synthetic solution products also showed some opaque particles, although the contaminants were in solution in the raw waste.

Most of the solidified products were not studied under the optical microscope.

Section 6

Effects of Solidification

The usefulness of a solidification process depends on the level of improvement effected by the process on the physical and chemical properties of raw wastes. In the following sections, the values of the physical and chemical properties measured for the solidified products are discussed, and compared with the raw waste properties where possible.

6.1 Physical Properties

All of the solidified products studied were solid monoliths. Thus, the first objective of solidification, namely the conversion of a liquid or semiliquid waste to a more easily handled solid, was met in all cases.

Since the solidification ingredients used were apparently similar for most of the products, the specific gravities of the different solidified products were consistent.

More variation between different solidified products was seen for two other physical properties: bulk density and moisture content. From a handling perspective, the differences in these properties were not important, but they result in large differences in the calculated porosities of the solidified products. Although drawing correlations between properties was outside the scope of this report, it can be surmised that less porous products have a lower hydraulic conductivity and provide greater physical resistance to leaching. Waste form durability may also be affected by moisture content and porosity.

The ranges of weight and volume change factors calculated for the different solidification processes were wide. These factors have a great impact on the cost of a solidification process because of the costs of solidification ingredients, transportation, and space in a landfill. No economic evaluation was carried out for the solidification processes investigated in this study, but economics are, of course, foremost in the minds of most waste generators and must be considered when evaluating a solidification process.

Hydraulic conductivity measurements ranged over four orders of magnitude, but with low accuracy. However, the maximum hydraulic conductivity for all solidified products was found to be 3×10^{-6} m/s. This is in the range of a silty sand, a medium that has relatively poor drainage characteristics, but is 1000 times more permeable than clay. Disposal of a waste form in a medium of lower hydraulic conductivity can result in groundwater flowing directly through the waste. The consequences of such groundwater flow on leaching have been described by Côté and Bridle (1987).

The unconfined compressive strength of the different solidified products varied widely. For disposal in a landfill, waste forms need only be strong enough to bear the weight of the overburden and land-moving equipment without losing their structural integrity. Although the low end of the observed range does not meet the 50 psi (345 kPa) guideline suggested by the United States Environmental Protection Agency (1986), the solidified product with the highest unconfined compressive strength is as strong as concrete. Since the median unconfined compressive strength was 1760 kPa (255 psi), the majority of the solidified products tested were more than strong enough for disposal in a landfill.

The solidified products were very different with respect to their abilities to endure cycles of freeze/thaw and wet/dry weathering. Some solidified products crumbled after one cycle, but almost half survived 12 freeze/thaw cycles, and almost three quarters survived 12 wet/dry cycles. Most disposal scenarios involve eventually protecting the waste form from natural freeze/thaw and wet/dry cycling, but since landfills are usually in operation for several years before they are sealed from the atmosphere, it is desirable for solidified wastes to exhibit a certain level of resistance to the elements. The importance of resistance to freeze/thaw and wet/dry cycling is dependent on the geographical location.

6.2 Chemical Properties

6.2.1 Equilibrium Extraction

The equilibrium extraction could not be applied to the Wastewater Technology Centre synthetic solution, as the contaminants were in solution. Table 34 shows the results of applying the equilibrium extraction procedure to the raw wastes, and also shows the median concentrations from equilibrium extraction testing of the solidified products (from Table 22).

Comparisons of the equilibrium extraction results for the raw wastes and the solidified products are discussed in the following sections.

Wastewater Technology Centre synthetic solution. Since the median pH of the extract for all the Wastewater Technology Centre synthetic solution solidified products was 12, the solubility of the metal contaminants was low, with median concentrations of arsenic, cadmium, chromium, and lead in the extract being less than 1 mg/L, which is clearly an improvement over having the metals in solution in the raw waste. A number of the solidification processes appear to have fixed the phenol to a minor extent, but the concentration in the extract from most solidified products was high, with a median of 310 mg/L.

Waterways Experiment Station synthetic sludge. For the extracts from all the Waterways Experiment Station synthetic sludge solidified products and the raw waste as well, the pH was around 12, resulting in median concentrations of cadmium, chromium, and nickel of less than 0.1 mg/L in the extracts from the solidified products and the raw waste. For mercury, however, the median concentration was 1 mg/L in the extracts from the solidified products, but 33 mg/L in the raw waste extracts. Thus, solidification appears to have improved the fixation of mercury.

Aluminum coil plating sludge. For the aluminum coil plating sludge solidified products, the extract pHs were somewhat lower than for the solidified products of the two synthetic wastes, but the pH for all solidified products was still greater than 8.6, whereas in the extract from the raw waste the pH was neutral. The concentrations of chromium, lead, and thallium were below detection in the raw waste extracts. In the extracts from the solidified products, chromium was above detection, but its concentration was still very low; most of the lead and thallium concentrations were below detection. The median concentration of arsenic in the solidified products was an order of magnitude lower than its concentration in the raw waste extract; however, for this waste, such a difference can be accounted for solely on the basis of dilution. For cyanide, the concentration measured in the raw waste extract was lower than those concentrations measured in the solidified products extracts. It is possible that more cyanide volatilized from the raw waste extract at pH 7 than from the high pH solidified product extracts. Overall, it appears that solidification did not substantially improve the chemical fixation of the contaminants.

Dredged sediment. Although the median pH in the extracts for the dredged sediments solidified products was near 12, the lowest pH was 7. The pH of the raw waste was also neutral. Median concentrations of chromium,

Table 34 Summary of Concentrations Measured in the Equilibrium Extraction for the Raw Wastes

Raw Waste	Parameter	Number of Samples	Concentration in Extract (mg/L) Raw Waste Average	Raw Waste Range	Solidified Product Median
WTC synthetic solution*	pH	-	-	-	12.0
	Arsenic	-	-	- -	0.25
	Cadmium	-	-	-	0.010
	Chromium	-	-	-	0.11
	Lead	-	-	-	0.47
	Phenol	-	-	-	310
WES synthetic sludge	pH	4	12.2	11.9 to 13.5	12.1
	Cadmium	4	ND	< 0.004	0.0014
	Chromium	4	0.14	0.04 to 0.46	0.083
	Nickel	4	ND	< 0.04	0.022
	Mercury	4	33	28.3 to 39	1.2
Aluminum coil plating sludge	pH	6	7.3	7.1 to 7.6	11.2
	Arsenic	4	0.020	0.01 to 0.138	0.0018
	Chromium	4	ND	< 0.016 to 0.07	0.28
	Lead	4	ND	< 0.012	0.018
	Thallium	4	ND	< 0.12	-
	Total cyanide	4	0.18	0.103 to 0.306	11.9
Dredged sediment	pH	2	7.7	7.7	11.9
	Chromium	4	ND	< 0.016	0.048
	Copper	4	ND	< 0.008 to 0.009	1.1
	Lead	4	ND	< 0.12	0.034
	Mercury	4	0.001	0.0003 to 0.003	0.00034
	Zinc	4	ND	< 0.01	0.059
	Polychlorinated biphenyls	5	ND	< 0.001	-
	Total organic carbon	5	26	17.8 to 30.9	251
Wood preservation waste soil	pH	6	4.4	4.1 to 5.1	11.7
	Arsenic	4	0.016	0.006 to 0.044	0.0013
	Lead	4	ND	< 0.12	0.0068
	Mercury	4	0.0027	0.00006 to 0.08	0.00018
	Pentachlorophenol	4	161	17.3 to 3576	97
	Polyaromatic hydrocarbons	4	ND	< 0.23	-
	Total organic carbon	2	389	347 to 436	442

Note: ND, not detected.

* The equilibrium extraction was not applied to this waste, as the contaminants were in solution.

lead, and zinc in the extracts from the solidified products were less than 0.1 mg/L, but were below detection in the raw waste extracts. The median concentration of copper was 1 mg/L in the extracts from the solidified products, but was below detection in the raw waste extract. However, for mercury, the median concentration in the extracts from the solidified products was an order of magnitude lower than its concentration of 0.001 mg/L in the extract from the raw waste. The concentrations of polychlorinated biphenyls in the extracts from the raw and solidified wastes were all below detection, but the median total organic carbon concentration was 250 mg/L in the extract from the solidified products and only 26 mg/L in the raw waste extract.

Solidification appears to have increased the solubility of all contaminants other than mercury, although the concentrations in the extracts remained low. The increase in metal solubility may have been a result of increasing the pH beyond the range of minimum solubility, or of oxidizing the waste form (Steneker et al., 1987).

Wood preservation waste soil. The median pH of the extracts from the wood preservation waste soil solidified products was near 12, whereas the lowest pH measured was 4.6, which was very close to the pH of the raw waste extract. The concentrations of the metals arsenic, lead, and mercury were very low in the raw waste extract (<0.1 mg/L), and about an order of magnitude lower in the extracts from the solidified products. Polyaromatic hydrocarbons were below detection in the extracts from the raw waste and the solidified products, and the median concentration of total organic carbon was similar for both the raw waste extract and the solidified product extracts. From the observed range of concentrations, it can be seen that some solidification processes appear to have achieved a slight reduction in the solubility of pentachlorophenol, although the median concentration found in the solidified product extracts is similar to the concentration measured in the raw waste extract.

6.2.2 U.S. EPA Toxicity Characteristic Leaching Procedure (TCLP)

Table 35 shows the results of applying the toxicity characteristic leaching procedure to the raw wastes, and the median concentrations resulting from applying the procedure to the solidified products (from Table 24). The procedure could not be applied to the Wastewater Technology Centre synthetic solution, as the contaminants were in solution.

The results of the toxicity characteristic leaching procedure for the solidified products were compared with the results for the raw waste. Also, as the procedure is a regulatory test, the contaminant concentrations measured in the leachates for the raw waste and solidified products were compared with the regulatory threshold levels of these contaminants for the procedure. The regulatory threshold levels are also listed in Table 35.

Wastewater Technology Centre synthetic solution. The leachates from the Wastewater Technology Centre synthetic solution solidified products had a median pH of 10, with a wider range of observed pHs than was seen in the equilibrium extraction because of the different capacities of the solidified products to neutralize the acid addition involved in the toxicity characteristic leaching procedure. The median concentrations of arsenic and cadmium were all less than 1 mg/L, but, again, the observed ranges were wider because of the wider range of leachate pHs. The median concentration of phenol in the extracts was 44 mg/L. This was lower than was observed in the equilibrium extraction because of dilution by the higher liquid-to-solid ratio of the toxicity characteristic leaching procedure.

The median concentrations of all metals in the solidified products leachates were lower than the regulatory threshold levels. For chromium and lead, the leachate concentrations from all solidified products were below the regulatory thresholds; however, for arsenic and cadmium, some leachate concentrations exceeded the threshold levels. Since the concentration of phenol in the majority of leachates exceeded the threshold level, most of the solidified products continued to exhibit hazardous characteristics after solidification.

For the Wastewater Technology Centre synthetic solution solidified products, the contaminant solubility was clearly a function of the pH of the extract. The ability of the waste form to neutralize acid addition, therefore, is a key characteristic for the fixation of metals.

Waterways Experiment Station synthetic sludge. For the toxicity characteristic leaching procedure leachate from the raw Waterways Experiment Station sludge, the average pH was 6. Although the pH was low, concentrations of cadmium, chromium, and nickel were less than 1 mg/L, whereas the concentration of mercury was about 3 mg/L. By comparison, for the leachates from all the solidified products, the median pH was 10. The majority of cadmium and nickel concentrations were below detection, and the median concentrations of chromium and mercury were less than 1 mg/L.

No threshold level for nickel is specified in the toxicity characteristic leaching procedure protocol, but the median concentrations of cadmium and chromium were lower than the

Table 35 **Summary of Concentrations Measured in the Toxicity Characteristic Leaching Procedure for the Raw Wastes**

			Concentration in Extract (mg/L)			
Raw Waste	Parameter	Number of Samples	Raw Waste Average	Raw Waste Range	Solidified Product Median	Regulatory Level (mg/L)
WTC synthetic solution*	pH	-	-	-	9.9	-
	Arsenic	-	-	-	0.17	5.0
	Cadmium	-	-	-	0.099	1.0
	Chromium	-	-	-	0.12	5.0
	Lead	-	-	-	0.046	5.0
	Phenol	-	-	-	43.9	14.4
WES synthetic sludge	pH	3	5.7	5.5 to 6.1	10.1	-
	Cadmium	3	ND	< 0.004 to 0.209	-	1.0
	Chromium	3	ND	< 0.016 to 0.831	0.052	5.0
	Nickel	3	ND	< 0.04 to 0.096	0.34	-
	Mercury	3	2.8	0.923 to 5.3	0.51	0.2
Aluminum coil plating sludge	pH	2	5.6	5.6	8.7	-
	Arsenic	2	0.001	0.001	0.0017	5.0
	Chromium	2	0.55	0.5 to 0.6	-	5.0
	Lead	2		0.003 to 0.005	-	5.0
	Thallium	2	ND	< 0.05	-	-
	Total cyanide	2	0.12	0.11 to 0.125	7.0	-
Dredged sediment	pH	1	5.0	-	7.8	-
	Chromium	1	0.09	-	0.020	5.0
	Copper	1	0.019	-	0.31	-
	Lead	1	ND	< 0.01	-	5.0
	Mercury	1	0.0006	-	0.0065	0.2
	Zinc	1	0.238	-	0.16	-
	Polychlorinated biphenyls	1	ND	< 0.00006	-	-
Wood preservation waste soil	pH	3	4.6	4.4 to 4.9	8.1	-
	Arsenic	3	0.0045	0.002 to 0.0077	-	5.0
	Lead	3	ND	< 0.12	0.011	5.0
	Mercury	3	0.011	0.0076 to 0.021	-	0.2
	Pentachlorophenol	3	33.7	0.021 to 6550	28	3.6
	Polyaromatic hydrocarbons	3	ND	< 1.2	-	-

Note: ND, not detected.

* The toxicity characteristic leaching procedure was not applied to this waste, as the contaminants were in solution.

threshold levels, for the solidified products and the raw waste, although the chromium concentrations for a few solidified products were greater than the threshold level. The mercury concentration was above the regulatory threshold level in the raw waste leachate and the majority of the solidified waste leachates, but it does appear that a few solidification processes succeeded in reducing all contaminant levels below the regulatory level specified for the toxicity characteristic leaching procedure.

A clear dependence of metal solubility upon pH was not as apparent for this waste. Since the sludge was aged, it was highly crystalline (see also Section 5.3), which may have decreased metal solubility at lower pHs.

Aluminum coil plating sludge. The raw waste toxicity characteristic leaching procedure leachate exhibited a pH of 5.6, whereas the median pH of the solidified waste leachates was 8.7. The metal concentrations were less than 1 mg/L in the raw waste leachates, and were not detected in the majority of solidified waste leachates. The average concentration of cyanide measured in the raw waste leachate was 0.1 mg/L, whereas the median concentration in the solidified waste leachates was 7 mg/L; these different levels may be a result of greater cyanide volatilization from the low pH raw waste extract.

Regulatory threshold levels for thallium and cyanide are not specified for the toxicity characteristic leaching procedure. Based on a comparison of the leachate levels with the threshold levels for arsenic, chromium, and lead, neither the raw waste nor the solidified products exhibited hazardous characteristics.

Dredged sediments. The pH of the raw waste toxicity characteristic leaching procedure leachate was 5.0, whereas the median pH of the leachates from the solidified products was 7.8. Median metal concentrations were less than 1 mg/L in the leachates from the solidified products and the raw waste, whereas polychlorinated biphenyl concentrations were all below detection.

Regulatory threshold levels for copper and zinc are not specified for the toxicity characteristic leaching procedure. Again, based on a comparison of the threshold and leachate levels of chromium, lead, mercury, and polychlorinated biphenyls, neither the raw waste nor the solidified products were leachate toxic.

The increase in contaminant solubility for the solidified dredged sediments, with respect to the raw waste, which was apparent for the equilibrium extraction, was not clear for the majority of solidified products in the toxicity characteristic leaching procedure, although it did appear to occur for some solidified products.

Wood preservation waste soil. For the solidified wood preservation waste soil, the median toxicity characteristic leaching procedure leachate pH was 8.1, whereas for the raw waste it was 4.6. Concentrations of arsenic, lead, mercury, and polyaromatic hydrocarbons were less than 0.1 mg/L in the raw waste leachates and below detection in the majority of solidified waste leachates. The median concentration of pentachlorophenol in the solidified waste leachates was 28 mg/L, and the average concentration of pentachlorophenol in the raw waste leachate was 34 mg/L. Thus, the majority of solidified products and the raw waste were leachate toxic, based on the regulatory threshold level of pentachlorophenol.

6.2.3 Acid Neutralization Capacity

Acid neutralization capacity was measured for the solidified Wastewater Technology Centre synthetic solution and the Waterways Experiment Station synthetic sludge only. Examination of the titration curves plotted for the different solidified products showed that the amount of acid necessary to bring the extract pH down to approximately 9 (where many metal hydroxides begin to become more soluble) varies from 2 to 10 meq per gram of dry waste. For comparison purposes, it can be calculated that 1 kg of a solidified product with an acid neutralization capacity of 2 meq/g can neutralize 2000 L of pH 3 groundwater. Therefore, it appears that even the solidified products with the lowest observed acid neutralization capacity have the potential to neutralize large quantities of acidic leachant, before the pore-water pH reaches a level where metal hydroxides will be dissolved.

6.2.4 Sequential Chemical Extraction

Table 36 shows the results of applying the sequential chemical extraction procedure to the raw wastes, and the median percentages extracted in each fraction for the solidified products (from Table 27). This test could not be applied to the Wastewater Technology Centre synthetic solution, as the contaminants were in solution.

Although the reproducibility of the method was poor (in the sense that no difference between solidified products could be observed), examination of the contaminant percentages extracted in the fractions of the sequential chemical extraction for the raw wastes and the solidified waste shows some trends with regard to contaminant distribution in the different fractions. These are discussed for the solidified products of each raw waste type.

Fractions A and B of the sequential extraction are the ion exchangeable fraction and the

Table 36 **Summary of Contaminant Percentages Extracted in the Fractions of the Sequential Chemical Extraction for the Raw Wastes**

Raw Waste	Parameter	Fraction	Number of Samples	Percentage in Fraction	
				Raw Waste Average	Solidified Product Median
WTC synthetic solution	*	-	-	-	-
WES synthetic sludge	Cadmium	A	2	0.005	0.11
		B	2	114.2	5.6
		C	2	2.11	82
		D	2	0.045	1.7
		E	2	0.0	0.13
		Total	2	116	132
	Chromium	A	2	0.055	0.099
		B	2	115.6	9.9
		C	2	2.2	107
		D	2	0.04	2.7
		E	2	0.01	0.49
		Total	2	118	138
	Nickel	A	2	0.0	0.089
		B	2	105.7	2.2
		C	2	2.0	123
		D	2	0.035	2.4
		E	2	0.0	0.28
		Total	2	108	129
	Mercury	A	2	1.5	0.57
		B	2	64.7	6.0
		C	2	19.0	17.8
		D	2	14.9	26
		E	2	0.17	3.3
		Total	2	101	118
Aluminum coil plating sludge	Arsenic	A	2	1.4	0.82
		B	2	0.87	5.5
		C	2	0.7	28
		D	2	0.96	17
		E	2	70.0	20
		Total	2	74	58
	Chromium	A	2	0.065	0.51
		B	2	46.7	41
		C	2	19.0	33
		D	2	39.6	6.6
		E	2	23.4	8.2
		Total	2	129	105
	Lead	A	2	0.07	-
		B	2	0.78	47
		C	2	0.49	19
		D	2	0.07	2.0
		E	2	9.4	36
		Total	2	11	186
	Thallium	A	2	0.0	-
		B	2	0.0	-
		C	2	0.0	-
		D	2	0.0	-
		E	2	0.0	-
		Total	2	0	0
Dredged sediment	Chromium	A	2	0.36	-
		B	2	23.6	31
		C	2	23.2	53
		D	2	12.0	17
		E	2	7.4	44
		Total	2	66	151
	Copper	A	2	3.0	8.1
		B	2	2.0	41
		C	2	17.9	41
		D	2	29.3	45
		E	2	12.1	41
		Total	2	64	171
	Lead	A	2	0.025	-
		B	2	88.8	79
		C	2	31.5	77
		D	2	3.4	11
		E	2	6.2	37
		Total	2	130	148
	Zinc	A	2	3.2	-
		B	2	38.2	55
		C	2	20.5	23
		D	2	7.9	7.1
		E	2	7.4	19
		Total	2	77	113

* The sequential chemical extraction was not applied to this waste, as the contaminants were in solution.

fraction extractable in a buffered pH 5 solution respectively. It has been suggested that the amount of contaminant measured in these fractions is the percentage of contaminant that is ultimately available for leaching under the harshest conditions likely to be encountered in a segregated landfill (i.e., a monofill, as opposed to a secure landfill, with an engineered barrier) (Kramer et al., 1988). The percentage of contaminant in fraction C may be available under the harsher, and more reducing, conditions of codisposal with municipal solid waste. Thus, the percentage of contaminant contained in fractions D and E is essentially unavailable for leaching. This interpretation of the results of the sequential chemical extraction should be kept in mind when examining the results obtained in this study.

Wastewater Technology Centre synthetic solution. For the majority of the Wastewater Technology Centre synthetic solution solidified products, more than 90% of the four metals (arsenic, cadmium, chromium, and lead) were found in fractions B and C together; fraction B tended to contain slightly more of these contaminants. Fraction A tended to contain less than 1% of these contaminants, and the remainder was distributed between fractions D and E. As the contaminants were in solution initially, solidification significantly improved their speciation.

Waterways Experiment Station synthetic sludge. In the untreated Waterways Experiment Station synthetic sludge, cadmium, chromium, and nickel were found almost exclusively in fraction B; more than 60% of the mercury was found in fraction B; and the remainder of the contaminants were primarily in fractions C and D.

However, in the solidified products, the median percentages of cadmium, chromium, and nickel show that the greatest portions of these contaminants were found in fraction C, whereas mercury was found in fractions C and D. For these solidified products in general, solidification appears to have improved the speciation of the metals with respect to their speciation in the raw waste; the metals also appear to be generally unavailable for leaching in a segregated landfill scenario.

Aluminum coil plating sludge. In the untreated aluminum coil plating sludge, the majority of arsenic was found in fraction E, whereas chromium was found to be distributed between fractions B, C, D, and E. Lead concentrations were very low, and thallium was below detection.

The median percentages in each fraction for the solidified products show arsenic to be distributed between fractions C, D, and E; chromium to be distributed primarily in fractions B and C; and lead to be distributed in fractions B, C, and E. It appears that, in this case, solidification has respeciated some of the contaminants, in some cases increasing their availability for leaching.

Dredged sediment. For the untreated dredged sediment, chromium was distributed mainly between fractions B, C, and D; copper was distributed between fractions C, D, and E; and lead and zinc were found in fractions B and C.

Based on the median percentages in each fraction for the solidified wastes, all four metals were distributed between fractions B, C, D, and E. In this case also, solidification appears to have respeciated some of the contaminants, although their availability for leaching was not necessarily decreased.

6.2.5 Dynamic Leach Test

The dynamic leach test was not performed for any of the raw wastes because it is applicable to monolithic samples only.

The apparent diffusion coefficient measured in the dynamic leach test is a function of the molecular diffusion coefficient of the contaminant, the matrix tortuosity, and the chemical containment of the contaminant.

Thus, the test measures both physical and chemical retardation of leaching. Contaminant mobility is a function of the apparent diffusion coefficient; the smaller the diffusion coefficient, the less mobile the contaminant. Since the leachability index is the negative logarithm of the diffusion coefficient, the greater the leachability index, the less mobile the contaminant.

The molecular diffusion coefficients for compounds in pure solution are typically on the order of 10^{-5} cm^2/s, i.e., a leachability index of 5. The leachability index for a contaminant in an untreated waste would be greater than this because of the contributing effects from other solutes and solid material in the waste. However, the leachability indices determined for the contaminants in the solidified products (see Table 29) can be compared with this number to estimate the improvement in immobilization achieved by solidification. A discussion of the contaminant mobilities observed for the solidified products of the three waste types tested using this method follows.

Wastewater Technology Centre synthetic solution. The leachability indices for arsenic, cadmium, chromium, lead, and phenol ranged over four to five orders of magnitude for the different solidified products. The median leachability indices measured for all four metals were greater than 12, but the median leachability index for phenol was only 7.7. In a number of cases, concentrations of cadmium, chromium, or lead were below detection in the leachate, which also indicated a very low contaminant mobility (i.e., a high leachability index).

It appeared that the mobility of the metals was very much reduced, but the mobility of phenol was decreased only slightly (with respect to its molecular diffusion coefficient) in the majority of these solidified products.

Aluminum coil plating sludge. The leachability indices observed for the different contaminants ranged over about four orders of magnitude. The median leachability index for chromium was 11.6. For lead, the median leachability index was 9.1, whereas the median leachability index for cyanide was only 8.4. Concentrations of arsenic and thallium were undetectable in the leachates, but for this waste, the likely cause is a low original concentration in the raw waste, rather than efficient immobilization.

Again, immobilization of the metals appeared to be more effective than immobilization of the cyanide. The low leachability indices for lead are probably an artifact caused by the low initial concentration of this contaminant in the solidified products (Stegemann and Côté, 1989).

Wood preservation waste soil. The median leachability index for pentachlorophenol was 8.5, with leachability indices ranging over three orders of magnitude. This was in the same range as the leachability indices for the organic compounds in the other two wastes; pentachlorophenol also appeared to be more mobile than the metal contaminants.

6.3 Micromorphological Properties

A tentative combined interpretation of the solidified product features observed using X-ray diffraction, scanning electron microscopy with energy dispersive X-ray analysis, and optical microscopy is given in Table 37, for the three waste types examined using these techniques. Since all of the techniques involved examination of minute samples, the conclusions drawn for each solidified product cannot be regarded as definitive.

Table 37 **Conclusions Derived from Micromorphological Characterization**

Industrial Participant ID Code	Possible Binder Components	Association of Contaminants with Matrix
WTC synthetic solution solidified products		
AAKC	Gypsum High alumina cement	Heavy metals in solid solution with gypsum
BABI	Portland cement Fly ash - type C Diatomaceous earth	Metals uniformly distributed, adsorbed to fly ash and diatoms
BAHE	Clay	Some reaction of phenol with binder
DEAI	Portland cement Gypsum Blast furnace slag	Metals uniformly distributed, adsorbed to fly ash surfaces
DEIF	Portland cement	Unknown
DGBF	Quartz filler Portland cement	Metals uniformly distributed
EAFI	Portland cement Fly ash - type C	Metals incorporated in crystal structure
EEBH	Portland cement Fly ash - type F	Metals uniformly distributed
EFBB	Portland cement Fly ash - type F Gypsum	Metals uniformly distributed
FAIG	Portland cement Fly ash - type F	Metals uniformly distributed
FCCI	Portland cement	Metals uniformly distributed
FFKK	Portland cement Fly ash - type C	Metals uniformly distributed
HDGF	Fly ash Gypsum Portland cement Quartz filler?	Metals uniformly distributed
IGIF	Portland cement Quartz filler?	Metals associated with quartz filler

Table 37 **Conclusions Derived from Micromorphological Characterization (cont'd)**

Industrial Participant ID Code	Possible Binder Components	Association of Contaminants with Matrix
WES synthetic sludge solidified products		
AIEI	Portland cement Gypsum?	Contaminants have respeciated from the sludge
AIKC	Portland cement Fly ash - type F	Sludge largely unreacted in matrix
BBIE	Portland cement Sand	Sludge largely unreacted in matrix
BDGE	Lime Gypsum	Sludge largely unreacted in matrix, but some contaminants in $CaSO_4$
BEDF	Portland cement Fly ash - type F	Sludge largely unreacted in matrix
DABB	Portland cement Gypsum Fly ash - type F	Sludge largely unreacted in matrix
EAHK	Portland cement Fly ash - type F K, Na, Cl?	Sludge largely unreacted in matrix
ECGH	Portland cement Fly ash - type F Gypsum	Some sludge unreacted in matrix, some contaminant interaction with binder
GBAB	Portland cement Fly ash - type F	Some sludge unreacted in matrix, some contaminant interaction with binder
GBFB	Portland cement	Some sludge unreacted in matrix, some contaminant interaction with binder
HGAB	Portland cement Fly ash - type C	Some sludge unreacted in matrix, some contaminant interaction with binder
KEAH	Portland cement Fly ash - type C	Sludge largely unreacted in matrix
KHEF	Portland cement	Sludge largely unreacted in matrix
KIAK	Portland cement	Sludge largely unreacted in matrix

Table 37 Conclusions Derived from Micromorphological Characterization (cont'd)

Industrial Participant ID Code	Possible Binder Components	Association of Contaminants with Matrix
Wood preservation waste soil solidified products		
ADDI	Portland cement	Unknown
BIDA	Portland cement	Unknown
BKDE	Portland cement Fly ash - type F?	Unknown
CDHB	Portland cement Gypsum	PCP largely microencapsulated
DEDB	Na, K, Cl?	Interaction of PCP with binder
DIDG	Gypsum	PCP not in gypsum
EIDA	Unknown (not portland cement)	Unknown
FDGI	Portland cement Fly ash - type F	Unknown
HBEG	Unknown	Unknown
HIKH	Polymer Gypsum	Some interaction of PCP with binder
IAHE	Portland cement Fly ash - type F Gypsum	Some interaction of PCP with binder
KBKC	Fly ash - type C Portland cement Gypsum	Some interaction of PCP with binder
KGAH	Unknown	Unknown

Note: PCP, pentachlorophenol.

Section 7

Conclusions and Recommendations

For each test, the usefulness of the measurement and the significance of the results obtained in this study are summarized in the following sections.

For each property, the observed range of values obtained for the different solidified products is also considered with respect to the interlaboratory reproducibility of the method. In cases for which this range of values was narrow, or when the interlaboratory variability was high, a test may not have differentiated between the different solidified products. However, such an observation does not preclude the usefulness of the test for purposes other than distinguishing between solidified products, or where a high degree of precision is not essential.

Although the interlaboratory variability frequently exceeded the intralaboratory variability, the latter is more representative of the inherent variability of a method. Therefore, the intralaboratory variability has been used to calculate a recommended number of replicates for obtaining a desired precision.

7.1 Raw Wastes

Analyses of waste aliquots and composite waste samples, repeated in the same laboratory and different laboratories, showed that interlaboratory and intralaboratory variabilities were similar, and that the wastes were homogeneous. The coefficients of variation were less than 50% for all contaminants whose average concentrations in the raw waste exceeded 100 mg/kg. For lower contaminant concentrations, the coefficient of variation was as high as 92%.

The reproducibility of the bulk density, total solids, and pH of the raw wastes appeared to be good.

7.2 Solidified Waste Properties

Chemical analysis of the solidified wastes did not always yield the same contaminant concentrations as were calculated using the contaminant concentrations in the raw waste and the weight change factors for the solidification processes. The difference was attributed to matrix effects, and it was assumed that the calculated contaminant concentration was the more accurate.

Liquid phases from solidification or pretreatment operations were submitted by the industrial participants with only five of the 69 solidified products. Metal levels in these liquid samples were typically less than 1 mg/L, whereas the phenol concentrations were several hundred milligrams per litre; in a practical scenario, additional treatment of these sidestreams would be required.

7.3 Physical Evaluation Test Methods

Table 38 summarizes the median and range of values measured for the physical properties of all the solidified wastes. The recommended number of test replicates is also presented. The average value obtained using this recommended number of replicates will differ from the true population mean of the property by the precision given in the final column of the table (at the 95% confidence level). Depending on whether the data distribution is normal or lognormal, the precision may be added or subtracted (±) to, or multiplied or divided (×/) by, the mean to determine the range in which the true value lies.

Table 38 Summary of Physical Testing Results

Test Method	Number of Products Tested	Median	Range			Recommended Number of Replicates	Precision
Bulk density (g/cm^3)	69	1.44	0.80	to	2.18	3	± 0.14
Specific gravity							
Wet method	69	2.48	1.75	to	3.53	4	± 0.28
Dry method	55	2.34	2.14	to	2.54	2	± 0.20
Moisture content (%,w/ww)	69	25.8	0.88	to	63.6	3	± 1.6
Hydraulic conductivity (m/s)	37	3×10^{-8}	7×10^{-10}	to	3×10^{-6}	4	630%
Unconfined compressive strength (kPa)	66	1 760	70	to	17 880	4	56%
Freeze/thaw weathering resistance	52						
Number of cycles to failure	29*	7	1	to	12.8	4	± 3
Percent weight loss after 12 cycles (w/dw)	23**	0.97	-0.17	to	8.1	4	± 4.0
Wet/dry weathering resistance	52						
Number of cycles to failure	14*	7	1	to	12.5	4	± 4
Percent weight loss after 12 cycles (w/dw)	38**	0.95	-8.6	to	28	4	± 3.5
Calculated Properties							
Weight change factor	69	1.79	1.14	to	6.04		
Volume change factor	69	1.55	0.96	to	4.04		
Porosity	69	0.57	0.16	to	0.81		

* Number of products that did not survive 12 cycles.
** Number of products that survived 12 cycles.

7.3.1 Engineering Properties

Bulk density, specific gravity, and moisture content are basic physical characteristics of a solidified waste that are also used in calculations for other parameters. There may also be correlations between these simple properties and a solidified waste's durability and leachability, although these were not investigated in this study.

The values for bulk density and moisture content varied relatively widely across the different solidified products. This variation was easily observable because of the good reproducibility of the methods between laboratories. Three replicates of each of these tests have been recommended for obtaining good precision.

The specific gravities of the different solidified products were similar to one another because of similarities in the solidification ingredients used. The dry specific gravity measurement method was substantially more precise than the wet method, but neither method showed clear differences between solidified products because of the narrow range of properties encountered. It may be noted that the two recommended replicates for the dry method will produce a more precise estimate of the specific gravity than the four replicates recommended for the wet method. Thus, the dry method is preferred.

The calculated porosities varied widely because of the different solidified waste bulk densities and moisture contents; these different porosities may have an impact upon the solidified wastes' durabilities and abilities to physically hinder leaching.

Two other calculated properties, the weight and volume change factors for the solidification processes, also varied over wide ranges, which could represent substantial differences in material, transportation, and landfilling costs.

7.3.2 *Hydraulic Conductivity*

A solidified waste's hydraulic conductivity, relative to that of the surrounding geology, determines whether leaching will occur by diffusion or advection (Côté and Bridle, 1987). Using the upper limit of the measured hydraulic conductivities as a conservative estimate, it was seen that solidified wastes are potentially more permeable than clays. Since field permeabilities are often orders of magnitude higher than laboratory permeabilities, this observation is of great practical importance in designing landfills for the disposal of these wastes.

Although the hydraulic conductivities observed for the different solidified products in this study appeared to range over four orders of magnitude, the differences between solidified products were not distinct because of poor interlaboratory reproducibility.

The recommended four replicates of this test result in a one order of magnitude precision.

Differences between specimens of the same solidified product appeared to be the primary cause of this variability, but development of a more accurate hydraulic conductivity measurement would be worthwhile. The "flow pump" measurement method described by Znidarcic et al. (1986), which measures the pressure required to induce flow through a sample, appears to be one technique worthy of further investigation.

7.3.3 *Durability*

The properties of unconfined compressive strength, and freeze/thaw and wet/dry weathering resistance, relate to the durability of the waste. Of course, the importance of a waste's resistance to freeze/thaw and wet/dry weathering is strongly dependent on the geographical location of the disposal site.

The range of unconfined compressive strengths was very wide, and the interlaboratory reproducibility was sufficiently good to allow the observation of clear differences between solidified products.

The majority of solidified products had an unconfined compressive strength much higher than the 50 psi (345 kPa) recommended by the United States Environmental Protection Agency. Four replicates of this measurement are recommended.

Most solidified products survived more than seven freeze/thaw and wet/dry weathering cycles. The weight loss of specimens that survived 12 freeze/thaw cycles was negligible, but the weight loss of specimens that survived 12 wet/dry cycles spanned the entire available range from 0 to 30% weight loss. Thus, it appears that the wet/dry test is more sensitive to differences between the different solidified products.

Even with four replicates, the precision for the number of cycles to failure is only three cycles for the freeze/thaw test and four cycles for the wet/dry test.

Since these tests are time and labour intensive, relationships between the weathering resistance and more easily measured properties should be explored. A possible correlation between the results of the two weathering tests should also be investigated.

7.4 *Chemical Evaluation Test Methods*

7.4.1 *Equilibrium Extraction*

The equilibrium extraction was designed to estimate the solubility of contaminants in the pore water of a solidified waste. The use of a distilled water leachant at a low liquid-to-solid ratio allows the waste to control the chemical environment. Crushing the waste to a fine powder results in relatively rapid attainment of steady-state conditions.

The contaminant concentrations found in the equilibrium extraction are summarized in Table 39.

In general, the equilibrium extraction results showed that metals, such as arsenic, cadmium, chromium, lead, nickel, mercury,

Table 39 Summary of Equilibrium Extraction Results

Parameter/Waste	Number of Products Tested	Number of Products Detected	Concentration (mg/L)				Number of Replicates	Precision
			Median	Range				
pH							4	+0.6
WTC synthetic solution	14	14	12.0	10.3	to	12.7		
WES synthetic sludge	14	14	12.1	11.7	to	12.8		
Aluminum coil plating sludge	13	13	11.2	8.6	to	12.5		
Dredged sediment	15	15	11.9	7.0	to	12.9		
Wood preservation waste soil	13	13	11.7	4.6	to	12.5		
Arsenic							4 11	150% 50%
WTC synthetic solution	14	14	0.25	0.010	to	1.2		
Aluminum coil plating sludge	13	3	0.0018	0.0010	to	0.0028		
Wood preservation waste soil	13	5	0.0013	0.00089	to	0.0023		
Cadmium							4 18	280% 50%
WTC synthetic solution	14	3	0.010	0.007	to	0.011		
WES synthetic sludge	14	1	0.0014	-				
Chromium							4 5	65% 50%
WTC synthetic solution	14	13	0.11	0.022	to	5.8		
WES synthetic sludge	14	14	0.083	0.012	to	2.4		
Aluminum coil plating sludge	13	13	0.28	0.021	to	1.18		
Dredged sediment	15	14	0.048	0.011	to	0.35		
Copper							4 11	150% 50%
Dredged sediment	15	12	1.1	0.14	to	4.0		
Lead							4 17	150% 50%
WTC synthetic solution	14	10	0.47	0.003	to	11.4		
Aluminum coil plating sludge	13	3	0.018	0.015	to	0.033		
Dredged sediment	15	6	0.034	0.021	to	0.089		
Wood preservation waste soil	13	1	0.0068	-				
Mercury							4 35	550% 50%
WES synthetic sludge	14	14	1.2	0.001	to	5.4		
Dredged sediment	15	5	0.00034	0.00021	to	0.00090		
Wood preservation waste soil	13	5	0.00018	0.00012	to	0.0019		
Nickel							4 7	90% 50%
WES synthetic sludge	14	10	0.022	0.008	to	0.099		
Thallium							-	-
Aluminum coil plating sludge	13	0	-	-				
Zinc							4 7	100% 50%
Dredged sediment	15	9	0.059	0.028	to	0.45		
Cyanide							4	100%
Aluminum coil plating sludge	13	13	11.9	0.027	to	325		
Pentachlorophenol							4 7	100% 50%
Wood preservation waste soil	13	13	97	5.3	to	317		
Phenol							4	10%
WTC synthetic solution	14	14	310	2.1	to	1880		
Polyaromatic hydrocarbons							-	-
Wood preservation waste soil	13	0	-	-				
Polychlorinated biphenyls							-	-
Dredged sediment	15	0	-	-				
Total organic carbon							4	50%
Dredged sediment	15	15	251	93	to	365		
Wood preservation waste soil	15	13	442	94	to	824		

copper, and zinc, have very low solubility in the high pH environment created by most solidified waste matrices. This is probably primarily a result of the formation of insoluble metal hydroxides (except in the case of arsenic, which is precipitated as basic calcium arsenite (Côté et al., 1987b)). Adsorption may also play a role in the immobilization of metals, particularly mercury. Only a few of the solidification processes investigated resulted in low concentrations of cyanide, phenol, and pentachlorophenol in the equilibrium extracts.

Comparing the equilibrium extract concentrations for the raw wastes and the solidified wastes showed that, under the conditions of the equilibrium extraction, solidification generally decreased the solubility of the contaminants in the Wastewater Technology Centre synthetic solution and the wood preservation waste soil, and mercury in the Waterways Experiment Station synthetic sludge and the dredged sediments. For the remaining contaminants, the extract concentrations for the solidified products were either similar or slightly increased with respect to the extract concentrations for the raw wastes. An increase in the equilibrium extraction concentration of contaminants in the solidified products, compared with that in the raw wastes, was particularly noticeable for the solidified dredged sediments, although leachate concentrations remained low.

Because of the very low concentration ranges under investigation, analytical reproducibility tended to be poor for the metals. Reproducibility was best for the organic contaminants that were not well immobilized and appeared in relatively high concentrations in the leachates. Results for contaminants whose concentrations in the raw waste were greater than 1000 mg/kg also tended to be more reproducible. Contaminants whose concentrations in the raw waste were less than 100 mg/kg were usually below detection in the leachates.

A consequence of the poor analytical reproducibility in the low concentration range, and the similarity of immobilization mechanisms in all the solidified products (which resulted in these low concentrations), was that differences between the different solidified products tended to be uncertain. However, the efficacy of the solidification processes was clear from these low concentrations, which showed that immobilization of the metals was successful.

Rather than recommending an exact number of replicates, Table 39 shows the precision for each contaminant, calculated for four replicates (based on the intralaboratory variability), and also the number of replicates required to produce a desired precision of ±50% (i.e., multiply or divide by 1.5). For four replicates, the calculated precision ranges from ±10 to 550% (i.e., ×/ 1.1 to 6.5); the number of replicates for a desired precision of ±50% ranges from 4 to 35. Based on this information, users of the test can estimate the number of replicates necessary to attain the precision they require. The number of replicates will vary based on the contaminants of interest and their own information concerning analytical variability, but a minimum of four replicates is recommended.

7.4.2 U.S. EPA Toxicity Characteristic Leaching Procedure (TCLP)

This is a regulatory test designed to assist with waste classification. Table 40 summarizes the concentrations measured in the extracts from this test for the different contaminants.

The results also showed that solidification was more successful in insolubilizing metals than organic compounds.

In comparison with the concentrations in the leachates from the equilibrium extraction, the ranges of contaminant concentrations in the toxicity characteristic leaching procedure tended to be wider because of the wider range that leachate pHs caused as a result of the

Table 40 Summary of Toxicity Characteristic Leaching Procedure Results

Parameter/Waste	Number of Products Tested	Number of Products Detected	Concentration (mg/L)				Number of Replicates	Precision
			Median	Range				
pH							4	+0.4
WTC synthetic solution	14	14	9.9	5.6	to	12.7		
WES synthetic sludge	14	14	10.1	7.8	to	12.5		
Aluminum coil plating sludge	13	13	8.7	5.2	to	10.7		
Dredged sediment	15	14	7.8	4.5	to	11.6		
Wood preservation waste soil	13	13	8.1	4.7	to	10.2		
Arsenic							4	280%
							19	50%
WTC synthetic solution	14	14	0.17	0.0087	to	36.4		
Aluminum coil plating sludge	13	1	0.0017	-				
Wood preservation waste soil	13	2	-	0.0032	to	0.031		
Cadmium							4	220%
							16	50%
WTC synthetic solution	14	8	0.099	0.003	to	31.5		
WES synthetic sludge	14	2	-	0.067	to	0.091		
Chromium							4	210%
							15	50%
WTC synthetic solution	14	9	0.12	0.030	to	1.34		
WES synthetic sludge	14	14	0.052	0.012	to	5.9		
Aluminum coil plating sludge	13	2	-	1.4	to	4.5		
Dredged sediment	15	4	0.020	0.0078	to	0.30		
Copper							4	76%
							6	50%
Dredged sediment	15	12	0.31	0.079	to	0.79		
Lead							4	1800%
							80	50%
WTC synthetic solution	14	7	0.046	0.0032	to	4.3		
Aluminum coil plating sludge	13	0	-	-				
Dredged sediment	15	0	-	-				
Wood preservation waste soil	13	1	0.011	-				
Mercury							4	180%
							13	50%
WES synthetic sludge	14	11	0.51	0.001	to	3.1		
Dredged sediment	15	5	0.0065	0.00028	to	0.013		
Wood preservation waste soil	13	0	-	-				
Nickel							4	160%
							11	50%
WES synthetic sludge	14	1	0.34	-				
Thallium							-	-
Aluminum coil plating sludge	13	0	-	-				
Zinc							4	150%
							11	50%
Dredged sediment	15	12	0.16	0.085	to	19.7		
Cyanide							4	30%
Aluminum coil plating sludge	13	13	7.0	1.8	to	29		
Pentachlorophenol							4	140%
							10	50%
Wood preservation waste soil	13	13	28	3.0	to	162		
Phenol							4	22%
WTC synthetic solution	14	14	44	0.073	to	144		
Polyaromatic hydrocarbons							-	-
Wood preservation waste soil	13	0	-	-				
Polychlorinated biphenyls								
Dredged sediment	15	0	-	-			-	-

different abilities of the solidified products to neutralize acid additions. The median contaminant concentrations in the toxicity characteristic leaching procedure extracts also tended to be slightly lower. For metals, this observation is attributable to the slightly lower median pH observed for the toxicity characteristic leaching procedure extracts; although most metals are much more soluble at low pHs than at high pHs, some metal species are amphoteric and tend to be more soluble at pH 12 than at pH 10. In some cases, however, the addition of acid in the toxicity characteristic leaching procedure decreased the extract pH sufficiently to increase metal solubility, demonstrating the importance of a product's acid neutralization capacity.

For organic compounds, the lower concentration in the toxicity characteristic leaching procedure extracts, compared with the equilibrium extraction, may be attributed to dilution by the procedure's higher liquid-to-solid ratio.

The median toxicity characteristic leaching procedure extract concentrations were compared with the regulatory threshold levels for the different contaminants, as specified in the procedure. This comparison showed that the majority of solidified Wastewater Technology Centre synthetic solution, Waterways Experiment Station synthetic sludge, and wood preservation waste soil products exhibited hazardous characteristics, whereas the aluminum coil plating sludge and dredged sediments were nonhazardous both before and after solidification. In the majority of hazardous solidified products, the problem contaminants were organic compounds and mercury.

Because of the wider concentration ranges observed in the toxicity characteristic leaching procedure extracts, differences between some solidified products were apparent, although most appeared similar because of high interlaboratory variability. Again, reproducibility was poor in the low concentration range, and better for higher contaminant concentrations.

As was done for the equilibrium extraction, the precision for four replicates was determined, as well as the number of replicates required for a desired precision of ±50% (i.e., ×/ 1.5). For four replicates, the precision for the different contaminants ranged from ±20 to 280% (i.e., ×/1.2 to 3.8), with the exception of lead, for which the precision was ±7800% (i.e., ×/ 19). The precision for lead was poor because the lead concentration in the extracts was extremely low (as a result of the pH), the lead concentration in the majority of extracts was below detection, and the median concentration in the remaining extracts was only 0.046 mg/L. For the other contaminants, the calculated number of replicates for a desired precision of ±50% ranged from 4 to 19. Again, a minimum of four replicates is recommended for this test.

The results of this regulatory test are intended for comparison with a threshold level. Exceeding the threshold level can result in the waste being classified as hazardous. Therefore, if the contaminant level and the threshold level differ by less than the precision with four replicates, more replicates may be required to increase the precision to determine whether the threshold level is truly exceeded or not. However, effective solidification of metals (usually by the production of hydroxides, with a high acid neutralization capacity) will generally result in concentrations that are unequivocally below the threshold limit.

7.4.3 Acid Neutralization Capacity

The acid neutralization capacity of a waste is a measure of the chemical environment in the waste, and relates particularly to its ability to immobilize metals, which tend to become soluble at pHs below 9. The pHs measured for different acid additions are summarized in Table 41.

Table 41 Summary of Acid Neutralization Capacity Results

Amount of Nitric Acid Added (meq/g of dry waste)	Number of Products Tested	pH Median	pH Range	Recommended Number of Replicates	Precision
				3	±2
0	33	12.2	10.7 to 13.2		
2.4	33	10.9	5.5 to 12.7		
7.2	31	7.2	0.8 to 11.3		
12	21	4.2	1.1 to 8.8		

The range of solidified product acid neutralization capacities was wide. Without exception, the solidified products were capable of neutralizing large quantities of acidic groundwater before reaching a pH where metal hydroxides would be dissolved. For example, 1 kg of a solidified waste with an acid neutralization capacity of 2 meq/g is capable of neutralizing 20 000 L of pH 4 groundwater.

Distinctions between solidified products became very clear at higher acid additions.

The test involves a series of 11 acid additions. For each of the 11 data points, three test replicates are recommended for a precision of ±2. However, plotting a titration curve using all 11 data points will increase the precision.

7.4.4 Sequential Chemical Extraction

The sequential chemical extraction procedure was designed to determine the speciation of metals, thus providing information about their availability for leaching.

Table 42 summarizes the sequential chemical extraction data for all the contaminants. For fractions B, C, D, and E, the amount extracted in the fraction ranged from near 0 to more than 100%, across the various solidified products. However, the median percentages extracted in the five fractions of the sequential chemical extraction, and the summation of the number of solidified products with maximum amounts leached in each fraction (Table 42), show that in the majority of solidified products the metals were speciated primarily in the second and third most available fractions (B and C). In general, this was also the case for the raw wastes. It appears that negligible amounts of all of the contaminants were speciated in the ion-exchangeable fraction (A) for the solidified wastes or the raw wastes, and usually only small amounts were found in fractions D and E.

It has been suggested that the amount contained in fractions A and B represents a conservative estimate of the fraction of the contaminant that is ultimately available for leaching in a segregated landfill (which is different from a secure landfill), whereas the amount contained in fraction C might be available under the more severe conditions of codisposal with municipal solid waste, and fractions D and E remain unavailable for leaching (Kramer et al., 1988). Thus, it appears that a large portion of the metals were speciated as unavailable for leaching in a segregated landfill in more than half the cases investigated.

Compared with the calculated contaminant concentrations for the solidified products, the total from all five fractions tended to overestimate the contaminant concentration in the samples by 2 to 86%, with the exception of arsenic in the aluminum coil plating sludge solidified products, which tended to be underestimated. Overestimation may be a

Table 42 Summary of Sequential Chemical Extraction Results

Contaminant/Waste	Number of Solidified Products with Largest Amount Extracted in Fraction				
	A	B	C	D	E
Arsenic	0	8	16	0	1
WTC synthetic solution	0	7	7	0	0
Aluminum coil plating sludge	0	1	9	0	1
Cadmium	0	16	10	2	0
WTC synthetic solution	0	11	2	1	0
WES synthetic sludge	0	5	8	1	0
Chromium	0	24	21	1	9
WTC synthetic solution	0	12	1	0	1
WES synthetic sludge	0	3	11	0	0
Aluminum coil plating sludge	0	6	5	0	1
Dredged sediment	0	3	4	1	7
Copper					
Dredged sediment	0	5	5	5	0
Lead	0	20	11	0	10
WTC synthetic solution	0	11	3	0	0
Aluminum coil plating sludge	0	5	1	0	6
Dredged sediment	0	4	7	0	4
Mercury					
WES synthetic sludge	0	4	10	0	0
Nickel					
WES synthetic sludge	0	1	13	0	0
Zinc					
Dredged sediment	0	13	2	0	0
TOTAL (for all metals)	0	91	104	8	20

result of contaminant carryover from one fraction to another, as a result of inadequate washing between steps. Total digestion and analysis for metals should always be conducted in parallel with the sequential chemical extraction.

For the two synthetic wastes, solidification clearly improved contaminant speciation; for the other two wastes tested, the aluminum coil plating sludge and the dredged sediments, contaminant speciation has been altered by solidification, although not necessarily improved.

Because of the similarity of immobilization mechanisms and poor interlaboratory reproducibility, no difference between solidified products was observed for this test. Further development of this test is necessary to refine the distinctions between fractions and reduce interlaboratory variability. Since the difference between speciation in fraction B and fraction C may be critical to interpreting contaminant speciation as being available or unavailable for leaching, a clear distinction between these two fractions is particularly important.

The calculations for the number of replicates for this test are based on the average intralaboratory variability for all the contaminants in all the fractions. The precision is ±150% (i.e., ×/ 2.5) the percentage extracted for four replicates; 10 replicates are required for a precision of ±50% (i.e., ×/ 1.5) the percentage extracted.

7.4.5 Dynamic Leach Test

The leachability index (i.e., the negative logarithm of an apparent diffusion coefficient) calculated from the amounts leached in each interval of the dynamic leach test provides a measure of a contaminant's mobility in the solidified waste matrix under diffusion control.

The results of the dynamic leach test are summarized in Table 43. Overall, it appeared that solidification decreased the apparent diffusion coefficient for metals by four to 11 orders of magnitude with respect to the molecular diffusion coefficient of the contaminant in pure aqueous solution. The apparent diffusion coefficient for organic compounds was decreased by only one to seven orders of magnitude.

Plotting the cumulative fraction leached against the square root of time, to verify the assumption of diffusion control inherent to this test, resulted in identifying four different leaching mechanisms: diffusion control, diffusion control following an initial resistance to leaching, diffusion control following an initial rapid washoff of contaminants, and linear release with time (for arsenic).

It was also shown that most specimens behaved as a semi-infinite medium, which was an assumption inherent to this test.

Comparison of the concentrations measured in the dynamic leach test leachates with the concentrations measured in the equilibrium extraction was carried out to verify the assumption of a maximum driving force for leaching, i.e., zero concentration at the sample surface. Concentrations in the equilibrium extracts were much higher than in the dynamic leach test leachates, unless the concentration in both tests was very near or below detection.

The reproducibility of this method was excellent, within and between laboratories, allowing for the observation of clear differences between solidified products. Only one replicate of this test is recommended, as a single test is an average of the results from seven or nine leaching intervals. A leachability index should not be calculated if data from more than one leaching interval are below detection.

7.5 Micromorphological Evaluation Methods

Conclusions drawn using X-ray diffraction, scanning electron microscopy with energy dispersive X-ray analysis, and optical microscopy are tentative because of the microscopic size of the samples examined using these techniques. Solidified products tend to be heterogeneous at a microscopic scale, so obtaining a representative sample is difficult. Consequently, these methods are more applicable as research tools for examining a few samples for well-defined features than as general methods for the overall investigation of a large number of solidified products. The observations made using the four micromorphological techniques are most useful in conjunction with one another.

X-ray diffraction and scanning electron microscopy data showed that portland cement and pozzolans (e.g., coal-burning fly ash, slag, etc.) were the main ingredients in many of the solidified products. However, many of the peaks registered in the X-ray diffraction

Table 43 Summary of Dynamic Leach Test Results

Parameter/Waste	Number of Products Tested	Number of LXs Calculated*	Leachability Index				Recommended Number of Replicates	Precision
			Median	Range				
Arsenic								
WTC synthetic solution	11	11	11.3	9.2	to	14.7	1	+0.7
Aluminum coil plating sludge	11	0	-	-				
Cadmium								
WTC synthetic solution	11	3	13.8	11.7	to	16.0	1	+0.6
Chromium								
WTC synthetic solution	11	8	12.2	10.3	to	13.3	1	+0.6
Aluminum coil plating sludge	11	11	11.6	9.6	to	13.0		
Lead								
WTC synthetic solution	11	9	12.7	9.1	to	14.5	1	+0.8
Aluminum coil plating sludge	11	9	9.1	7.1	to	10.2		
Thallium								
Aluminum coil plating sludge	11	9	-	-			-	-
Cyanide								
Aluminum coil plating sludge	11	11	8.4	6.3	to	11.6	1	+0.4
Pentachlorophenol								
Wood preservation waste soil	12	11	8.5	7.7	to	10.7	1	+0.4
Phenol								
WTC synthetic solution	11	11	7.7	6.4	to	11.8	1	+0.4

Note: LX, leachability index.

* The leachability index could not be calculated if the contaminant concentration in the leachate from more than one interval was below detection.

patterns remained unidentified, as did some crystalline phases seen using scanning electron microscopy. Interference between peaks for different elements was a continual problem for the energy dispersive X-ray analysis. All of these phenomena varied depending on the waste and the solidification process.

From the energy dispersive X-ray analysis, it appeared that the metals present in the Wastewater Technology Centre synthetic solution were uniformly distributed in the solidified products. Some interaction between the phenol in this waste and the matrices was noted in the X-ray diffraction patterns.

In the case of the Waterways Experiment Station synthetic sludge, scanning electron microscopy, X-ray diffraction, and optical microscopy showed that the sludge particles (approximately 40 μm in diameter) were encapsulated by the matrices in the majority of the products. From the energy dispersive X-ray analysis, it appeared that the metals remained primarily associated with the original sludge particles, although minor respeciation may have occurred.

For the wood preservation waste soil solidified products, the interaction between the pentachlorophenol and the matrix remained largely unknown as the soil matrix dominated the scanning electron microscopy and X-ray diffraction observations.

7.6 Recommendations

7.6.1 Sources of Error

Although analytical methods were agreed upon and strictly specified, identical methods were not used in all of the laboratories. The development of more extensive and standardized methods in all three laboratories would have reduced an important source of interlaboratory error.

Backlogging of analytical samples for different time periods in all three of the heavily loaded laboratories could also have increased the variability. Such a practice should be avoided.

Except for unconfined compressive strength measurements, the curing period of the solidified products before testing also varied considerably between the three laboratories.

This was probably not a significant source of variation in the results from testing, but adherence to the standard curing period of 56 days is desirable.

7.6.2 Applicability of Test Methods

Each of the test methods performed and analyzed in this study was designed to measure a different solidified waste property. These solidified waste properties are significant only in the context of the proposed disposal environment. For disposal in a particular scenario, some properties are of greater importance than others. Therefore, the selection of tests, and performance criteria for these tests, depends upon the properties of importance in a chosen disposal scenario. As a follow-up to this study, a document that recommends performance criteria for four different disposal and utilization scenarios has been prepared (WTC, 1990).

The analysis of test method reproducibility and the range of properties encountered in this study showed that the tests differed in their abilities to distinguish between different solidified products, although all appeared capable of producing a measurement of the property of interest. Test method reproducibility must be taken into account when designing a testing program, or interpreting results from testing.

7.6.3 Further Work

The analysis and interpretation of the results of this study were limited by the objectives originally outlined because of the large amount of data generated. However, great potential for continued analysis of these data exists, particularly with regard to relationships between different solidified waste properties.

The test methods investigated in this study represent the state of the art at the time of the study's initiation, and cover a wide range of solidified waste properties. However, test method development is an evolutionary process; as the technology and understanding of waste characteristics develop, the development of these and other test methods should be continued.

In particular, continued development of test methods to examine the following topics is desirable: contaminant availability, waste stability in environments of different oxidation/reduction potentials, immobilization of organic contaminants, and biodegradability.

The results of this study also indicate that containment of even relatively small quantities of organic compounds in primarily inorganic waste types is not easily effected by solidification. Development of solidification formulas for enhanced containment of organics is essential if this technology is to be used for site cleanup, or the treatment of complex wastes with traces of organic contaminants, without pretreatment.

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Appendix A

Industrial Participant Information

Note: Appendices B to J, as listed in the Table of Contents, are available upon request.

A&A
Anderson Tank Service
10471 No. 3 Road
Richmond, British Columbia
V7A 1W8
Canada

Contact: Mr. W. Tilbury

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.

Chemfix Technologies Inc.
P.O. Box 1572
1675 Airline Highway
Kenner, Louisiana
70063
U.S.A.

Contact: Dr. P. Lo

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.

Chemical Waste Management Inc.
150 West 137th Street
Riverdale, Illinois
60627
U.S.A.

Contact: Mr. Jesse Connor

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.

Dansk Beton Teknik A/S
Hellerupland Alle 21
Hellerup, Denmark
DK-2900

Contact: Mr. Anders Henrichsen

Raw Waste	Solidified Product Code
WTC synthetic solution	DGBF
WES synthetic sludge	GBFB
Aluminum coil plating sludge	AIHF
Dredged sediment	HBBK
Wood preservation waste soil	CDHB

Note: See information sheets that follow.

Process Information Sheet

WTC Synthetic Solution

Company name: Dansk Beton Teknik A/S

Was the waste treated before solidification? No

Mass of waste solidified: 2688 g

Additive		Amount Added (g)
1.	Cement	5000
2.	Sand	3000
3.	Microfiller	1000
4.	Water and plasticizer	632

Mixing time (min): 5

Process Information Sheet

WES Synthetic Sludge

Company name: Dansk Beton Teknik A/S

Was the waste treated before solidification? No

Mass of waste solidified: 18 000 g

Additive		Amount Added (g)
1.	Cement	18 000
2.	Sand	2 000
3.	Microfiller	5 800
4.	Water and plasticizer	4 200

Order of addition: One third of the cement was added and mixed before the rest

Mixing time (min): 5

Process Information Sheet

Aluminum Coil Plating Sludge

Company name: Dansk Beton Teknik A/S

Was the waste treated before solidification? No

Mass of waste solidified: 6880 g

Additive	Amount Added (g)
1. Cement	2500
2. Sand	2500
3. Microfiller + superplasticizer	1000

Mixing time (min): 5

Process Information Sheet

Dredged Sediment

Company name: Dansk Beton Teknik A/S

Was the waste treated before solidification? No

Mass of waste solidified: 8 265 g

Additive	Amount Added (g)
1. Porous material	10 657
2. Cement	2 044
3. Microfiller	545
4. Sand	3 736
5. Water and plasticizer	1 000

Order of addition: The porous material was added first

Mixing time (min): 5

Process Information Sheet

Wood Preservation Waste Soil

Company name: Dansk Beton Teknik A/S

Was the waste treated before solidification? No

Mass of waste solidified: 4285 g

Additive	Amount Added (g)
1. Cement	5000
2. Microfiller	1000
3. Sand	1000
4. Water and plasticizer	1000

Mixing time (min): 5

Dofasco Incorporated
Box 460 1330
Burlington Street East
Hamilton, Ontario
L8N 3J5
Canada

Contact: Mr. Murray Greenfield

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.

Harmon Environmental Services
5221 Militia Hill Road
Plymouth Meeting, Pennsylvania
19462
U.S.A.

Contact: Mr. W. Webster

Raw Waste	Solidified Product Code
WTC synthetic solution	HDGF
WES synthetic sludge	ECGH
Aluminum coil plating sludge	HHHC
Dredged sediment	AEHH
Wood preservation waste soil	EIDA

Note: No further information was revealed.

Geotechnical Research Centre
McGill University
817 Sherbrooke Street West
Montreal, Quebec
H3A 2K6
Canada

Contact: Dr. R. Yong

Raw Waste	Solidified Product Code
WTC synthetic solution	Not solidified
WES synthetic sludge	Not solidified
Aluminum coil plating sludge	Not solidified
Dredged sediment	DBCC
Wood preservation waste soil	Not solidified

Note: No further information was revealed.

Peterson Contractors Inc.
104 Blackhawk
Reinbeck, Iowa
50669
U.S.A.

Contact: Mr. Bill Heithoff, Jr.

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.

The PQ Corporation
280 Cedar Grove Road
Lafayette Hill, Pennsylvania
19444
U.S.A.

Contact: Ms. E. Davis

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.

Solidtek Systems Inc.
P.O. Box 888
5371 Cook Road
Morrow, Georgia
30260-0888
U.S.A.

Contact: Mr. J. Slemmer

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.

Stablex Canada Inc.
760 Boulevard Industriel
Blainville, Quebec
J7C 3V4
Canada

Contact: Mr. P. Grenier

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.

Tricil Limited
89 Queensway West
Mississauga, Ontario
L5B 2V2
Canada

Contact: Mr. P. Suppa

Raw Waste	Solidified Product Code
WTC synthetic solution	FAIG
WES synthetic sludge	AIEI
Aluminum coil plating sludge	EABB
Dredged sediment	DBFK
Wood preservation waste soil	IAHE

Note: See information sheets that follow.

Process Information Sheet

WTC Synthetic Solution

Company name: Tricil Limited

Was the waste treated before solidification? No

Mass of waste solidified: 7.5 L

Additive		Amount Added
1.	Pickling acid	5.2 L
2.	Slag	1120 g
3.	Lime	1390 g
4.	Slag	2000 g
5.	Cement	1200 g

Mixing time: (1+2+3) 1 hour, (4+5) 1/2 hour

Other important process details: Filtration of 6.2 L after 1,2,3

Process Information Sheet

WES Synthetic Sludge

Company name: Tricil Limited

Was the waste treated before solidification? No

Mass of waste solidified: 6 000 g

Additive		Amount Added (g)
1.	H_2SO_4 solution	19 640
2.	Slag	1 140
3.	Lime	2 210
4.	Slag	5 000
5.	Cement	4 100

Mixing time: (1+2+3) 1 hour, (4+5) 1/2 hour

Other important process details: Filtration of 13 L after 1,2,3

Process Information Sheet

Aluminum Coil Plating Sludge

Company name: Tricil Limited

Was the waste treated before solidification? No

Mass of waste solidified: 5000 g

Additive		Amount Added (g)
1.	Slag	2000
2.	Lime	650
3.	Cement	1500
4.	Water	3000

Mixing time: (1+2+3+4) 1/2 hour

Process Information Sheet

Dredged Sediment

Company name: Tricil Limited

Was the waste treated before solidification? No

Mass of waste solidified: 8000 g

Additive		Amount Added	
1.	Hydrochloric acid	2.4	L
2.	Slag	480	g
3.	Lime	900	g
4.	Slag	2500	g
5.	Cement	750	g

Mixing time: (1+2+3) 1 hour, (4+5) 1/2 hour

Process Information Sheet

Wood Preservation Waste Soil

Company name: Tricil Limited

Was the waste treated before solidification? No

Mass of waste solidified: 2500 g

Additive		Amount Added (g)
1.	Water	1500
2.	Slag	900
3.	Lime	500
4.	Slag	350
5.	Cement	1200

Mixing time: (1+2+3+4+5) 1/2 hour

United States Gypsum Company
700 North Highway 45
Libertyville, Illinois
60048
U.S.A.

Contact: Mr. K. Watkins

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.

Velsicol Chemical Corporation
2603 Corporate Avenue, Suite 100
Memphis, Tennessee
38132
U.S.A.

Contact: Mr. George Harvell

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: Solidification additives included portland cement and coal-burning fly ash.

West-Paine Laboratories
7979 GSRI Avenue
Baton Rouge, Louisiana
70820
U.S.A.

Contact: Dr. P. West

Raw Waste	Solidified Product Code
WTC synthetic solution	Not revealed
WES synthetic sludge	Not revealed
Aluminum coil plating sludge	Not revealed
Dredged sediment	Not revealed
Wood preservation waste soil	Not revealed

Note: No further information was revealed.